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AFML-TR-77-71

EXPLORATORY DEVELOPMENT OF CORROSION INHIBITING PRIMERS

Northrop Corporation
Aircraft Group
Hawthorne, California 90250

May 1977

TECHNICAL REPORT AFML-TR-77-71

Final Report for Period May 1976 — March 1977

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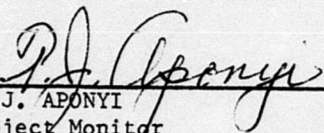


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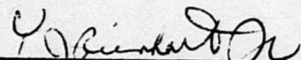
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This technical report has been reviewed and is approved for publication.


T. J. APONYI
Project Monitor

FOR THE COMMANDER


T. J. REINHART, JR., Chief
Composite and Fibrous Materials Branch
Nonmetallic Materials Division

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The objective of this program was to develop a corrosion inhibiting primer suitable for adhesive bonding which can also serve as corrosion protection for detail aluminum parts. The developed primer must be adaptable to an automated application method. The program utilized the electropainting application method and investigated the polyester, acrylic, and epoxy electropainting resins. The anodic and the cathodic electropainting processes were evaluated. The cathodic application was shown to be the preferred method when compared to the interfacial oxide effects produced by the anodic applications. Results of these studies produced a cathodically applied modified epoxy electropainter, C-5301. Assessment tests of the C-5301 primer show acceptable corrosion protection capability and compatibility with current state-of-the art paint primers and topcoats.

Adhesive bonding test results have shown the C-5301 primer superior in some applications and the control BR-127 primer superior in others. The electrodeposited C-5301 primer was shown to be far superior in reproducibility and uniformity. The developed C-5301 electropainter is readily adaptable to automated processing methods and can provide uniform, reproducible films which are cost effective.

CONCLUSIONS AND RECOMMENDATIONS

The developed C-5301 contract primer performs satisfactorily for corrosion protection of aluminum substrates. The adhesive bonding performance data for the C-5301 primer looks promising and indicates further development is required to provide a universal electropainter superior in all respects to the state-of-the-art hand-sprayed primers.

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PREFACE

This report was prepared by the Northrop Corporation, Aircraft Group, Hawthorne, California under USAF Contract F33615-76-C-5301. The contract work was performed under Project No. 7340, Task No. 734002, and administered under the direction of the Air Force Materials Laboratory. The program monitor was Mr. T.J. Aponyi (MBC) of the Composites and Fibrous Materials Branch of the Nonmetallic Materials Division, AFML.

Mr. S. L. Diener served as the Principal Investigator on this program. Other Northrop personnel who made major contributions in this research program were R. E. Herfert, J. D. Mackey, and T. B. Pollard.

The Sherwin-Williams Company personnel who made major contributions in this research program were B. C. Watson and S. J. Mels.

The contractor's report number is NOR 77-69. This report covers work from 1 May 1976 through 28 February 1977.

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SUMMARY

For the past five years, Northrop, under independent research funds, has been actively investigating the electropainting of aluminum substrates. An anodically applied polyester electropainter was developed and approved by the Air Force for corrosion control of aluminum detail parts on the Northrop F-5 series aircraft. A feasibility study to apply electrodeposited primers for adhesive bonding of aluminum alloy substrates was completed which confirmed compatibility of electropainters with current state-of-the-art epoxy film adhesives. The feasibility study also indicated wide variations in stressed durability response by different electropaint formulations. The development of a universal corrosion inhibiting electropainter, i.e., serve as a preparation for adhesive bonding and as corrosion protection for aluminum alloy parts, then continued with Air Force Materials Laboratory sponsorship under Contract F33615-76-C-5301. This contract was divided into five phases. Results obtained on each phase are summarized below.

PHASE I — PRIMER SYSTEM DEVELOPMENT

An improved electropainter was sought which was compatible with 250F and 350F curing adhesives and provided a more durable adhesive bond than the current state-of-the-art electropainters. The polyester, acrylic, and epoxy electropainting resin systems were investigated. The initial development effort was on the polyester system which Northrop had gained considerable experience with in precontract studies. The initial attempts to promote acceptable durability response incorporated a strontium chromate inhibitor into the electropaint formulation. No significant improvement was realized and the chromated polyester wedge test performance on FPL surface treated substrates was poor. It was determined that the maximum usable strontium chromate concentration was 1.5%. Higher concentrations presented stability problems. Formulation modifications with the polyester resin crosslinker (catalyst) to promote maximum crosslinking and ether linkages were not successful relative to wedge test performance. The acrylic electropainting resin formulation was evaluated and screening tests showed an incompatibility with the 250F curing epoxy film adhesive, FM-123. Screening tests of a 400F curing modified epoxy electropainter provided acceptable

wedge test results and showed compatibility with the current state-of-the-art 250F curing film adhesives. Further formulation development showed improved performance utilizing a titanium dioxide pigmented formulation versus a nonpigmented formulation. Final formulation development efforts reduced the modified epoxy electroprimer cure schedule to 345F for 30 minutes.

PHASE II — PROCESS OPTIMIZATION

The objective of Phase II was to optimize the electropriming application process to promote maximum adhesive bond durability. The study was initiated by an investigation of the anodic electropriming process. Application potential and application time variables were studied. Scanning electron microscopy (SEM) analyses showed that an FPL etch surface treatment oxide increases in thickness with an increase in application potential. Wedge test evaluations showed the oxide thickness increase was detrimental to wedge test performance. It was determined that wedge test performance of anodically electroprimed substrates could be significantly improved by low voltage deposition from a high solids bath. Wedge test performance equivalent to the BR-127 control primer could not be achieved with the anodic deposition process.

Investigation of the cathodic electrodeposition process clearly demonstrated superiority of cathodic electropriming over the anodic electrodeposition process relative to wedge test performance and effects on the interfacial primer/substrate oxide. A process variable study provided application parameters for the C-5301 contract primer.

PHASE III — PRIMER PERFORMANCE ASSESSMENTS

An assessment was made of the C-5301 primer and cathodic application process developed in Phases I and II. The developed contract primer was shown to be compatible with various paint primers and topcoats and provided acceptable corrosion protection for aluminum substrates. Adhesive bonding tests demonstrated the C-5301 contract primer superior in performance in some of the required mechanical tests and the BR-127 control primer superior in others.

PHASE IV — COST AND SCALE-UP ASSESSMENT

An engineering assessment of the electroprime process accomplished by Northrop determined the feasibility and cost effectiveness of installing an automated electroprime line for corrosion protection of detail aluminum parts. The engineering assessment

recommended the procurement and installation of an electropriming facility with a projected savings of 2 million dollars based on a seven-year amortization period with a 2.2-year payback.

The comparative costs of hand spraying the BR-127 adhesive primer versus the automated electroprime application of the developed contract primer show a potential annual savings of \$425,000. An investment of approximately \$800,000 would be required to implement a completely automated C-5301 cathodic electropriming facility.

PHASE V — UNIFORMITY AND REPRODUCIBILITY ASSESSMENT

The comparative evaluation of the uniformity and reproducibility of the C-5301 contract primer versus the hand-sprayed BR-127 adhesive primer show the C-5301 electroprimer is far superior in these respects. The study shows the C-5301 primer film to be reproducible, uniform, and independent of operator technique. Electroprimer films applied 0.00018 inch in thickness were reproducible within 0.00001 inch and uniform to within ± 0.00003 inch.

SECTION I

INTRODUCTION

The current state-of-the-art in preparing aluminum alloy substrates for adhesive bonding is to chemically produce a stable boehmite oxide on the aluminum surface, followed by oven drying and hand-spray application of an adhesive primer. The four limitations associated with the surface preparation method are well known and are identified as: (1) the produced boehmite oxide is subject to change during the time between oven drying and primer application, (2) the produced oxide is subject to contamination between oven drying and primer application, (3) the hand-sprayed application of the adhesive primers is limited in its ability to provide uniform reproducible films relative to film thickness and dispersion of corrosion inhibiting agents, and (4) the current state-of-the-art method is costly.

The required solutions to the defined problem areas are the subject of this contract program. This program developed a cathodically deposited modified epoxy electrophimer suitable for application by cost effective, automated methods.

The electrophimering process incorporates the application of the primer as part of the surface treatment and eliminates the intermediate oven drying and transport currently required. Consequently, the boehmite oxide is not subject to change or contamination. The intrinsic character of the electrophimering process provides a uniform, reproducible film independent of operator technique or material stratification. The electrophimering process is cost effective, since it is readily adaptable to automated processing methods.

The primary objectives of this contract effort were to:

- a. Develop a corrosion inhibiting primer for adhesive bonding aluminum alloys which is reproducible and cost effective to apply.
- b. Develop a primer which will be compatible with 250F curing structural adhesives and if possible with the 350F curing adhesives. Further, the developed primers shall be compatible with aircraft paint primers and topcoats.

To meet these objectives, the required contract effort was to:

- a. Develop an electroprimer which would be compatible with 250F curing adhesives.
- b. Optimize the electropriming application process for maximum bond durability.
- c. Comparatively evaluate the resultant electropriming system with the best state-of-the-art hand-sprayed adhesive primer, BR-127.
- d. Evaluate the uniformity, reproducibility, and cost effectiveness of the developed system.

SECTION II

TECHNICAL DISCUSSION

PHASE I — PRIMER SYSTEM DEVELOPMENT

Background

Northrop has been actively engaged in the study of electrodeposited organic materials for the past five years. Intrinsic to the electropriming process are certain aspects which make it uniquely attractive to the exploratory development of corrosion inhibiting primers. These aspects are:

1. Total coverage, including recesses and "blind" areas.
2. Uniform film deposits independent of operator technique.
3. Cost effective, since the process is readily automated.
4. Ecologically effective by coating from a water-base system.

The electrodeposition of organic materials is basically accomplished by solubilizing a resin system and electrically depositing the resin from a water solution onto a conductive substrate. The cathodic film deposition mechanism is illustrated in the Appendix A. Since the organic resin is nonconductive, the deposition is forced onto conductive areas of the "work-piece" until the entire surface has been insulated. The thickness of the deposited film is regulated by the applied potential with a maximum thickness of approximately 1 mil. The film deposition time is approximately 60 seconds.

Northrop selected the electrodeposition process to exploit the intrinsic advantages of the process in the development of a corrosion inhibiting primer. Although it was not the intent of the program to develop a new surface treatment for bonding, considerable attention had to be given to the effects on the interfacial oxide produced by electropriming on the preliminary surface treatment, i.e., FPL etch or 10 volt phosphoric acid anodize. Therefore, considerable effort was expended in the process optimization study.

Northrop, under independent research funding, studied the electro-deposition of polyester resins systems to aluminum substrates for corrosion control. Successful

completion of this effort produced a corrosion inhibiting primer qualified to stringent company specifications and approved by the Air Force for application to the F-5 aircraft. (NAI 1330 ELECTRODEPOSITED CORROSION INHIBITING, FLUID RESISTANT PRIMER.) See Appendix B.

Initial tests relative to bondability of the polyester electroprimed aluminum substrates produced equivalent or higher mechanical strengths when compared to conventional FPL etched specimens. The results of these tests were particularly encouraging relative to failure modes of the test specimens in that all cohesive failures were realized. These tests substantiated the compatibility of electrodeposited primers with a standard 250F curing modified epoxy adhesive and showed the adhesive to be the weakest link in the bonded joint. However, a cursory study of the electroprimed and adhesive bonded aluminum tensile specimens, subjected to durability testing in a saltwater environment and loaded to 50 percent of the ultimate strength, demonstrated a wide variation in the performance of different electroprime formulations.

It was believed that a durable polyester primer system for bonding could be developed through modification of the primer with a corrosion inhibiting agent and the durability of the system could be promoted by enhancing the resin/oxide "mixture" zone. The "mixture" zone depth can be controlled by varying the electrical application parameters during anodic application of the resin. Indeed, the initial primer modification incorporating chromates did enhance the durability of the system as assessed by the 3M durability (stress rupture) test. The first attempt to incorporate a corrosion inhibitor into the polyester resin system utilized lead chromate at a 15 percent level (dry weight basis). Based on work accomplished in earlier programs, it was established that strontium chromate at a 2-5 percent level was sufficient for corrosion inhibition. The 2-5 percent strontium chromate concentration was utilized for the initial investigations to develop a universal corrosion inhibiting primer, i.e., serve as a preparation for adhesive bonding and as a corrosion protection for detail aluminum alloy parts.

The feasibility study to apply electrodeposited primers for adhesive bonding aluminum alloys was completed by Northrop under independent research and development funds. The study confirmed compatibility of electroprimers with current state-of-the art epoxy film adhesives, but also indicated wide variations in stress durability test response by different electroprime formulations. The development of a universal corrosion inhibiting primer then continued with Air Force Materials Laboratory sponsorship under Contract F33615-76-C-5301 awarded to Northrop on 1 May 1976. The

program requirements were basically defined as follows:

1. Phase I — Primer System Development
Formulate an electrodepositing primer for aluminum alloy substrates with the objective of optimizing the durability aspects of a bonded system.
2. Phase II — Process Optimization
Determine the optimum process application parameters for applying the developed electroprimer.
3. Phase III — Primer Performance Assessment
Utilizing the developed primer and the process parameters evolved in Phases I and II, assess the overall performance of systems with various adhesives and paint topcoats.

The work accomplished under the AFML Contract is presented and discussed in the balance of this section.

Development

This contract development effort was accomplished with the Sherwin Williams Company as the resin formulator. Eighteen individual formulations were developed and screened for film formulation properties and basic tank stability. Northrop, as the Prime Contractor, evaluated the trial formulations relative to bonding performance and evaluated the data to direct reformulation and modification efforts required by the resin formulator. A definition of the candidate formulations is tabulated in Appendix C.

Based on the enhancement of bonding properties noted by chromate additions in precontract evaluations of the polyester electropriming resin system, the initial pursuit in the resin development was to incorporate the strontium chromate inhibitor.

The following strontium chromate formulations were prepared:

<u>FORMULATION SERIES</u>	<u>FORMULATION ID</u>	<u>PERCENT SrCr_2O_7</u>
A - Polyester	SA 4185	0.5
A - Polyester	SA 4186	1.0
A - Polyester	SA 4187	1.5

The initial attempts to chromate the formulations at the higher strontium chromate concentrations were not possible due to settling (instability) of the system at the higher concentrations. Although the polyester resin system tolerated levels of

lead chromate up to 15 percent, it was determined that a maximum level for the strontium chromate was about 1.5 percent. This same low tolerance level for strontium chromate was also experienced with the subsequent modified epoxy electropriming resins systems. With concentration levels of strontium chromate greater than 1.5 percent, the resin system coagulates in the bath and precipitates out even under vigorous agitation. The subsequent film deposits are "grainy" and irregular. The lower (0.5 - 1.5 percent) levels of SrCr_2O_7 permit a uniform application of the resin film.

Since the electrical application parameters for each formulation varies, the first assessment for each formulation was the determination of the electrical parameters to deposit a uniform film in the range of 0.1 - 0.3 mil in thickness. To determine the electrical application requirements for the strontium chromated samples, specimens of 2024-T3 bare and 7075-T6 bare were prepared for electropriming by FPL etching and 10-volt phosphoric acid anodizing. FPL etched panels were coated with SA 4187 by increasing the voltage to 35 volts in ten seconds and holding this potential for 30 seconds. This produced an average cured film thickness of 0.6 mil. The 10 volt phosphoric acid anodized panels were also coated with SA 4187 by increasing the potential to 55 volts in ten seconds and holding this potential for 30 seconds. This produced an average cured film thickness of 0.8 mil. Since the thicknesses produced were considered excessive relative to previous tests, lower voltages and times were employed to provide the thickness required. The required coating voltage potentials and times for the SA 4185, SA 4186, and SA 4187 polyester trial formulations were determined by laboratory tests and were as follows:

<u>SURFACE TREATMENT</u>	<u>APPLICATION POTENTIAL (volts)</u>	<u>APPLICATION TIME (seconds)</u>	<u>FILM THICKNESS (mil)</u>
FLP Etch	20	20	0.2
10 Volt Phosphoric Acid Anodize	50	10	0.3

The initial screening test series run on the strontium chromated polyester resin system utilized the wedge test. This test used both the FPL etch and the 10 volt phosphoric acid anodize surface preparation. Specimens were bonded with the FM-123-2 adhesive. The test specimen configuration is defined in Appendix D. Results of these tests are presented in Table 1.

TABLE 1. WEDGE TEST RESULTS OF
STRONTIUM CHROMATED POLYESTER ELECTROPRIMERS
CURED AT 275F FOR 30 MINUTES

FORMULA- TION, ID.	STRONTIUM CHROMATE CONTENT (%)	WEDGE TEST CRACK EXTENSION (IN.)			
		SURFACE PREPARATION			
		10 VOLT ANODIZE		FPL ETCH	
		1 HOUR	24 HOUR	1 HOUR	24 HOUR
SA 4185	0.5	0.1	0.2	1.0	1.7
SA 4186	1.0	0.1	0.1	1.4	1.8
SA 4187	1.5	0.1	0.1	1.0	1.5
SA 4191 control	0	0.1	0.2	1.1	1.8

- Notes: 1. Reported values are averages of 5 determinations.
2. Exposure conditions - 120F, 95% RH.
3. Primer cured at 275F for 30 minutes.
4. 0.125 inch thick 7075-T6 bare adherends.

This wedge test series was run to define the necessity and level of strontium chromate when compared to the control SA 4191 primer without chromate. No distinct enhancement in wedge test performance was discernible after testing. The polyester electroprimer on the 10 volt phosphoric acid anodized surface treatment was equivalent to the BR-127 baseline data. However, this same primer over the FPL surface treatment showed a gross difference in performance when compared to a BR-127 baseline.

The reasons for the large differences in crack extension results were that the primer did not develop a full cure and the FPL etch surface treatment provided a more sensitive test. The normal cure schedule for the polyester system when utilized for corrosion control alone is 275F for 30 minutes.

The wedge tests were then rerun on primed substrates which had been cured at 290F for 30 minutes. Although a significant improvement on all three primers was noted, no differentiation between chromated versus non-chromated primers could be established. The wedge test data is presented in Table 2.

Analysis of the wedge test data led to the conclusion that the polyester primer was sensitive to water (hydrolytic reversion). This required that further polymer development be undertaken. From the known fact that polyether linkages are less susceptible to hydrolytic reversion compared to polyesters, an attempt was made to modify the formulation to promote ether linkages. Formulations SA 4188 and SA 4190 produced. Formulation SA 4188 contained an increased concentration of crosslinking* (catalyst) agent and SA 4190 utilized an experimental crosslinking agent.

*See Appendix E for definition of terms.

Wedge tests were conducted on these two formulations with primer cure temperatures of 275F and 290F. Results of these wedge tests are presented in Table 3.

TABLE 2. WEDGE TEST RESULTS OF
STRONTIUM CHROMATED POLYESTER ELECTROPRIMERS
CURED AT 290F FOR 30 MINUTES

FORMULA- TION, ID.	STRONTIUM CHROMATE CONTENT (%)	WEDGE TEST CRACK EXTENSION (IN.)			
		SURFACE PREPARATION			
		10-VOLT ANODIZE		FPL ETCH	
		1 HOUR	24 HOUR	1 HOUR	24 HOUR
SA 4185	0.5	0.1	0.2	0.7	1.2
SA 4186	1.0	0.1	0.1	0.7	1.1
SA 4187	1.5	0.1	0.3	0.4	1.0
SA 4191 control	0	0.1	0.1	0.8	1.1

- Notes:
1. Reported values are averages of 5 determinations.
 2. Exposure conditions - 120F, 95 % RH.
 3. Primer cured at 290F for 30 minutes.
 4. 0.125 inch thick 7075-T6 bare adherends.

TABLE 3. WEDGE TEST RESULTS OF
INITIAL POLYESTER MODIFICATIONS

FORMULA- TION, ID	MODIFICATION	CURE TEMP. F.	WEDGE TEST CRACK EXTENSION (IN.)	
			SURFACE PREPARATION	
			10-VOLT ANODIZE	FPL ETCH
			24 HOUR GROWTH	24 HOUR GROWTH
SA 4188	Increased concen- tration of cross- linker	275	0.2	1.6
		290	0.2	1.2
SA 4190	Experimental crosslinker	275	0.2	2.7
		290	0.1	1.6
SA 4191	Control (Standard Polyester formulation)	275	0.2	1.8
		290	0.1	1.1

- Notes:
1. Reported values are averages of 5 determinations.
 2. Exposure conditions - 120F, 95% RH.
 3. 0.125-inch thick 7075-T6 bare adherends.

Test results showed that at both primer cure temperatures the primer with the standard crosslinker was more durable. An infrared analysis (IR) was performed on the two crosslinker variation formulations and compared with the control formulation. A less durable bond in the wedge test was realized even though the IR analysis indicated a reduction in hydroxyl content.

To comparatively evaluate the five polyester primer modifications, a series of stress rupture tensile specimens were prepared utilizing the configuration shown in Appendix D. The FPL etch and 10 volt phosphoric acid anodize were used prior to electropriming. These specimens, bonded with FM-123-2 adhesive, were loaded to 50% of their room temperature ultimate strength and exposed to condensing humidity at 140F. The test results did not differentiate any differences in the primer formulations. It was believed that the test conditions were too severe. The test results were coordinated with the AFML program monitor and the test parameters were changed as follows: (1) the exposure temperature was lowered to 120F and (2) the center hole, as defined by the "Raab" configuration, was omitted. A second series of stress rupture specimens were prepared using the FPL etch and the 10 volt phosphoric acid anodize and bonded with the FM-123-2 adhesive. A set of BR-127 primed panels served as controls. Results of this test series are presented in Table 4. Here again, we note a gross difference in the stress rupture test results between the 10 volt phosphoric acid anodize pretreatment and the FPL pretreatment. The difference factor is approximately 10:1 and is comparable with the wedge test results of the primers applied over the two surface treatments.

TABLE 4. STRESS RUPTURE TEST RESULTS OF
POLYESTER ELECTROPRIMERS

FORMULATION ID (1) (POLYESTER)	STRESS RUPTURE TEST (2) (3) (4)	
	SURFACE PREPARATION	
	10-VOLT ANODIZE	FPL ETCH
	(HOURS TO FAILURE)	(HOURS TO FAILURE)
SA 4185	365	44
SA 4186	221	40
SA 4187	254	23
SA 4188	202	38
SA 4190	214	34
SA 4191	324	36
Control		
BR-127	472	412
Control		

- Notes:
1. Reference Appendix C table for formulation and definition.
 2. Standard 3M stress durability test apparatus.
 3. Average of 3 determinations - 120F, condensing humidity, 40% RT ultimate load.
 4. 0.063-inch thick 7075-T6 bare adherends

In conjunction with results concurrently emanating out of the Phase II development effort, it became apparent that a cathodically applied material should be investigated. Therefore, a modified epoxy formulation (SA 4521) which the Sherwin-Williams Company had formulated for other commercial applications was made available for initial screening purposes.

The initial tests on the new cathodic system determined the electrical application parameters to deposit a 0.1 - 0.3 mil uniform film. An application potential range of 25 to 90 volts and application times from 1 to 30 seconds were studied. Results of this study indicated that the optimum deposition parameters were 50 volts for five seconds. The initial wedge test panels utilized the FPL etch preparation, since this surface treatment indicated a more sensitive response in previous tests. The electroprimed substrates were cured at 400F for 30 minutes to develop full cure of the primer and bonded with the FM-123-2 adhesive. The wedge test results are presented in Table 5.

TABLE 5. WEDGE TEST RESULTS OF
CATHODIC EPOXY ELECTROPRIMER SA-4521

FORMULATION ID	WEDGE TEST (4) CRACK EXTENSION (IN.)	
	1 HOUR	24 HOUR
SA 4521-1	0.06	0.15
SA 4521-2	0.03	0.11
SA 4521-3	0.04	0.11
SA 4521-4	0.05	0.11
SA 4521-5	0	0.05

- Notes: 1. Exposure conditions - 120F, 95% RH.
2. Primer cured at 400F for 30 min.
3. 0.125-inch thick 7075-T6 bare adherends.
4. FPL etch surface treatment

These test results were encouraging, confirming the direction taken relative to cathodic application. A second formulation for test of the same basic resin systems modified with a different crosslinker (catalyst) was screened. This formulation (SA 4522) was developed to lower the cure temperature requirement of the primer system. Upon "let-down" (adding the required amount of water to electropriming consistency), the resin system coagulated and could not be utilized for test.

To determine the time and temperature required to fully cure the SA 4521 primer, seven 7075-T6 bare panels were FPL etched, electroprimed, and cured between

200F and 400F and checked for cure sufficiency with an MEK rub test. Results indicated that a cure temperature of 350F would be required. Therefore, wedge test and tensile shear test panels of 7075-T6 bare with the electroprimer cured at 350F, 375F, and 400F were prepared with FM-123-2 adhesive and tested. Test results are presented in Table 6.

TABLE 6. TENSILE SHEAR AND WEDGE TEST RESULTS
OF SA 4521 ELECTROPRIMER

ELECTROPRIMER CURE CYCLE	WEDGE TEST (5) CRACK EXTENSION (IN.) (24-HOUR GROWTH)	TENSILE SHEAR TEST ULTIMATE PSI (4)(5)	
		RT	180F
30 minutes at 350F	1.3	4410	2960
30 minutes at 375F	0.3	4210	2890
30 minutes at 400F	0.2	4220	2920

- Notes: 1. Test results are averages of 4 specimens.
2. Wedge test exposure - 120F, 95% RH.
3. 0.125-inch thick 7075-T6 bare adherends.
4. All failures were cohesive.
5. FPL etch surface preparation.

Although the failure modes of the tensile shear test specimens were all 100% cohesive, the wedge test results indicated that a cure temperature of 400F is required for fully curing this primer formulation.

Since the 400F cure temperature is considered excessive, the Sherwin-Williams Company cathodically applied acrylic electropriming resin system, with a 325F cure, was selected for screening as a candidate primer. Two cathodic acrylic electroprime formulations were supplied for evaluation. The SA 4708 is a non-chromated formulation and the SA 4753 contains 3% strontium chromate on a dry weight basis. The screening test results are presented in Table 7.

The failure modes on the tensile shear specimens indicated an incompatibility of the acrylic electroprimer with the FM-123-2 adhesive system in that 20% to 80% adhesive failures were realized. At the 180F tensile test temperature, the results were scattered. Based on these test results when compared to the results of the modified epoxy primer, a decision was made to work exclusively with the cathodically applied epoxy electroprimer.

The Sherwin-Williams Company, through crosslinker development, formulated a lower cure temperature (375F cure) modified epoxy electroprimer. This formulation, SA 4777, was identical in all respects to the basic SA 4521 modified epoxy

TABLE 7. WEDGE AND TENSILE SHEAR TEST RESULTS
OF CATHODIC ACRYLIC ELECTROPRIMERS

Formulation ID	Wedge Test Crack Extension (In.)		Tensile Shear (3) Ultimate Strength (PSI) ⁽⁶⁾			
	Surface Preparation		Surface Preparation			
	10-Volt Anodize	FPL ETCH	10-V Anodize		FPL ETCH	
	24-Hour Growth	24-Hour Growth	RT	180F	RT	180F
SA 4708 Non-Chromated Cathodic Acrylic Electroprimer	0.25	1.62	4470	1740	4420	2480
SA 4753 Chromated Cathodic Acrylic Electroprimer	0.35	1.36	3870	2000	4430	2390

- Notes: 1. Reported values are averages of 5 determinations.
2. Exposure conditions - 120F, 95% RH.
3. Notched shear configuration. (See Appendix D.)
4. Primer cured at 325F for 30 minutes.
5. 0.125-inch thick 7075-T6 bare adherends.
6. Combination adhesive/cohesive failures.

electroprimer with the exception of the crosslinker. A series of wedge tests on 7075-T6 bare confirmed the cure temperature of 375F for 30 minutes.

A primer formulation performance review was then held with the Sherwin-Williams Company to provide specific direction for the final formulation effort. Since some benefit from chromating the electrodepositing resin system had been realized and pigmenting could be detrimentally affecting the primer system, i.e., providing a moisture propagation path, the following plan for the final formulation effort by Sherwin-Williams Company was implemented:

1. Provide pigmented and unpigmented resin formulations.
2. Provide chromated and nonchromated resin formulations.
3. Provide an alternate inhibitor.

Following these guidelines, formulations were prepared and are defined in the Appendix C. Basically, they were as follows:

SA 4824 - The SA 4777 modified epoxy containing 1.5% strontium chromate.

SA 4825 - The SA 4777 modified epoxy containing 1.5% strontium chromate but without TiO₂ pigment.

SA 4826 - The SA 4777 modified epoxy containing 1.5% NALZIN SC-1* inhibitor.

SA 4827 - The SA 4777 modified epoxy containing 1.5% NALZIN SC-1 inhibitor but without TiO₂ pigment.

The latest generation of modified epoxy primers were formulated to evaluate chromated versus nonchromated electropimers and also pigmented versus nonpigmented electropimers. The series also contained a catalyst which should have provided a full resin cure at 325F. It was later determined that the 325F for 30 minutes cure schedule was not sufficient to completely cure the electropimer resin system. The comparative wedge test results did, however, make the necessary definitions in performance of pigmented versus nonpigmented electropimers and also chromated versus Nalzin inhibitors. The first test series compared the TiO₂ pigmented versus the nonpigmented versions of the modified epoxy primer. Wedge test specimens of 7075-T6 bare were prepared and tested. These tests results are reported in Table 8. The wedge test data show the pigmented version of the modified epoxy electropimer was superior to the nonpigmented version. Apparently the titanium dioxide pigment functions as a binder and promotes better adhesion of the primer to the substrate.

TABLE 8. WEDGE TEST RESULTS OF
PIGMENTED VERSUS NONPIGMENTED
CATHODIC EPOXY ELECTROPRIMERS

FORMULATION IDENTIFICATION	WEDGE TEST RESULTS			
	SURFACE PREPARATION			
	10-VOLT ANODIZE		FPL ETCH	
	CRACK EXTENSION (IN.)		CRACK EXTENSION (IN.)	
	1-HR GROWTH	24-HR GROWTH	1-HR GROWTH	24-HR GROWTH
SA 4826 Pigmented Epoxy Primer	0.1 Cohesive Failure	0.1 Cohesive Failure	0.6 Adhesive Failure	1.1 Adhesive Failure
SA 4827 Non- Pigmented Epoxy Primer	0.1 Cohesive Failure	0.1 Cohesive Failure	1.3 Adhesive Failure	2.0 Adhesive Failure

- Notes: 1. Reported values are averages of 5 specimens.
2. Exposure conditions - 120F, 95% RH.
3. All specimens bonded with FM-123 adhesive.
4. Primer cured at 325F for 30 minutes.
5. 0.125-inch thick 7075-T6 bare adherends.

*NALZIN SC-1 is a zinc phospho oxide complex, product of National Lead Corporation.

The Nalzin inhibitor was incorporated on an experimental basis. No data relative to its performance on nonferrous alloy substrates is available. The Nalzin inhibitor was recommended by the Sherwin-Williams Company based on their experience with electroprimers on ferrous substrates.

A retest of the SA 4826 wedge test was performed at a primer cure temperature of 400F. Results of these tests are presented in Table 9.

TABLE 9. WEDGE TEST RESULTS OF CATHODIC EPOXY ELECTROPRIMERS

FORMULATION ID	SPECIMEN	WEDGE TEST RESULTS CRACK GROWTH (IN.) ⁽⁵⁾	
		1 HOUR	24 HOUR
SA 4826 - Pigmented Epoxy Primer	1	0.07	0.12
	2	0.05	0.17
	3	0.12	0.34
	4	0.07	0.34
	5	0.11	0.31

- Notes: 1. Exposure conditions - 120F, 95% RH.
 2. All specimens bonded with FM-123 adhesive.
 3. Primer cured at 400F for 30 minutes.
 4. 0.125-inch thick 7075-T6 bare adherends.
 5. FPL etch surface preparation.

This test data confirms the test primer, SA 4826, does provide acceptable wedge test response, but the primer must be cured above 325F for 30 minutes.

The test formulation series SA 4825 and SA 4826, which contained 1.5% strontium chromate, required additional effort to balance the film coalescing solvent level. After various voltage, time, and current application variable techniques were tried, it was determined that adequate film coalescence could not be achieved. Therefore, no further attempts were made to chromate the modified epoxy electroprimers.

It was established that the cathodically applied modified epoxy electrophimer was compatible with a standard epoxy film adhesive, i.e., FM-123-2, and wedge test performance comparable to the BR-127 baseline primer could be achieved. Therefore, the selection for the overall assessment of Phase III of this program was made.

The modified epoxy electrophimer would be pigmented with titanium dioxide and incorporate the Nalzin SC-1 inhibitor. An exact cure schedule would have to be developed to ascertain the minimum time and temperature required for the selected primer to be comparable to the BR-127 adhesive primer. The contract primer, designated

C-5301, was compounded and wedge test specimens of 7075-T6 bare with an FPL surface treatment were prepared. This primer was cured over a temperature range of 325F to 400F in 20F increments. Test results are presented in Table 10.

TABLE 10. WEDGE TEST RESULTS OF
C-5301 PRIMER AT VARIOUS CURE TEMPERATURES

PRIMER ID	PRIMER CURE TEMPERATURE ⁽³⁾ (°F)	WEDGE TEST CRACK EXTENSION (IN.) ⁽²⁾⁽⁵⁾	
		1 HOUR	24 HOUR
BR-127 (Control)	250	0.07	0.25
Modified Epoxy Electro- Primer, Contract Primer (C-5301)	325	0.83	0.19
C-5301	345	0.09	0.19
C-5301	365	0.08	0.19
C-5301	385	0.07	0.21
C-5301	400	0.05	0.19

- Notes: 1. Reported values are averages of 5 test specimens.
2. Standard 120F, 95% relative humidity exposure.
3. Contract primer cured 30 minutes at indicated temperature.
4. 0.125-inch thick 7075-T6 bare adherends.
5. FPL etch surface preparation.

At all primer cure temperatures above 325F for 30 minutes, the contract primer performed equivalent to the BR-127 control. After 24 hours the C-5301 primer crack extension values shown in Table 10 are superior to the BR-127 control. This test lacks definition at these lower crack extension growth values, and the comparative performances must be assessed by the stress rupture test. The stress durability assessment was accomplished in Phase III of the program utilizing both the 10 volt phosphoric acid anodize surface treatment and the FPL etch surface treatments.

A second study made relative to the C-5301 primer cure schedule was to extend the cure time at the lower cure temperatures. The cure temperatures of 325F and 345F were examined and the cure time extended from 30 minutes to 60 minutes. Results of this study are presented in Table 11 and show the C-5301 primer provides acceptable wedge test results when cured for 60 minutes at 325F.

In summary, the Phase I development effort screened three candidate resin types; the anodically applied polyester, the cathodically applied acrylic, and the cathodically applied modified epoxy resin systems.

**TABLE 11. WEDGE TEST RESULTS OF
C-5301 ELECTROPRIMER AT VARIOUS CURE TIMES**

PRIMER ID	PRIMER CURE TEMPERATURE (°F)	PRIMER CURE TIME (MINUTES)	CRACK EXTENSION (IN.)	
			1 HOUR	24 HOUR
C-5301	325	30	0.83	1.19
	325	60	0.06	0.19
	345	30	0.09	0.19
	345	45	0.09	0.24
	345	60	0.06	0.15

- Notes:
1. Reported values are averages of 5 test specimens.
 2. Exposure conditions - 120F, 95% RH
 3. All specimens FPL etched and bonded with FM-123 adhesive.
 4. 0.125-inch thick 7075-T6 bare adherends.

The cathodically applied modified epoxy was selected to be utilized for the overall assessment tests in Phase III of the development program. This formulation, designated C-5301 electroprimer, is defined as follows:

Base Resin	Modified Epoxy
Crosslinker (catalyst)*	Sherwin-Williams Company Proprietary
Coalescing Solvent*	Sherwin-Williams Proprietary
Pigment *	Titanium Dioxide
Corrosion Inhibitor*	Nalzin SC-1, 1.5%
Dye*	Iron Yellow
Cure Requirements	345F for 30 Minutes
Application	30 Volts - 15 Seconds
	(Cathodic)

*See Appendix E for definition of terms

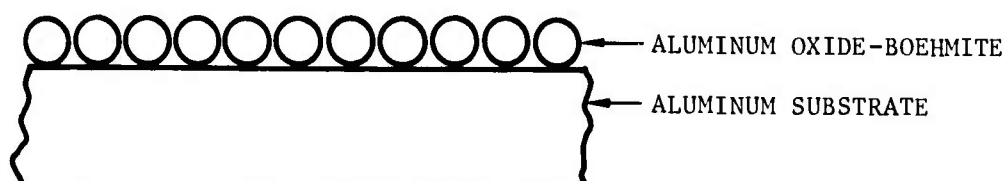
PHASE II - PROCESS OPTIMIZATION

Northrop, during investigations to apply a polyester electroprimer anodically for corrosion control, determined that the interfacial oxide produced by the electroprime process was the boehmite oxide. This is the desirable aluminum oxide form for bonding and is the specific oxide form produced by the standard FPL etch and 10 volt phosphoric acid anodize surface preparations on aluminum for adhesive bonding. Further instrumental analysis, i. e., Auger, determined that the electroprime process

did indeed codeposit a mixture zone of boehmite oxide and resin represented pictorially in Figure 1.

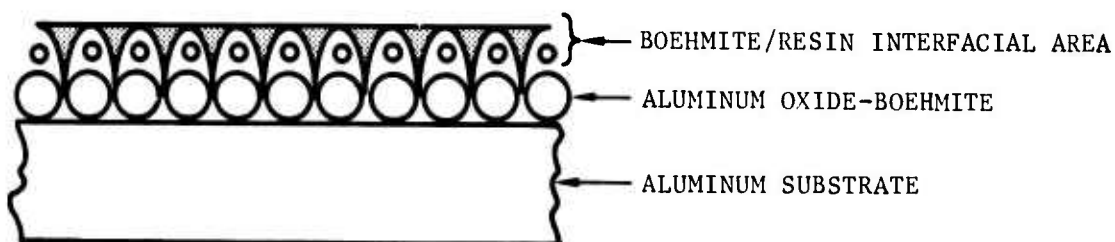
STEP I

PROCESS INITIALLY DEPOSITS A LAYER OF BOEHMITE



STEP II

BUILD-UP OF BOEHMITE CONTINUES WITH CO-DEPOSITION OF RESIN PRIMER PROMOTING ADHESION



STEP III

THE FINAL DEPOSITION OF RESIN PRIMER SEALS THE OXIDE - OXIDE/PRIMER LAYERS TO PROMOTE DURABILITY

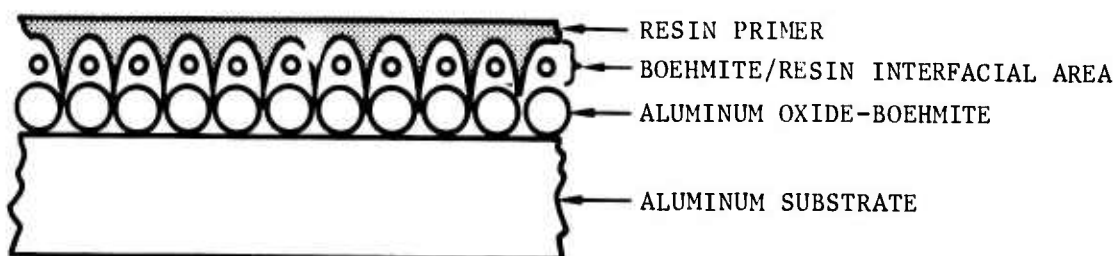


FIGURE 1. ANODIC ELECTROPRIMING MECHANISM

With the anodic electropriming process as the program starting point, it was believed that maximum durability of a bonded joint could be achieved by optimizing this interfacial area. To accomplish this goal, a stable boehmite oxide should be deposited on the surface of the aluminum substrate, maximizing the penetration depth of the

"inter-mixture" zone of boehmite and organic resin, followed by complete envelopment of the substrate with the electrodeposited primer.

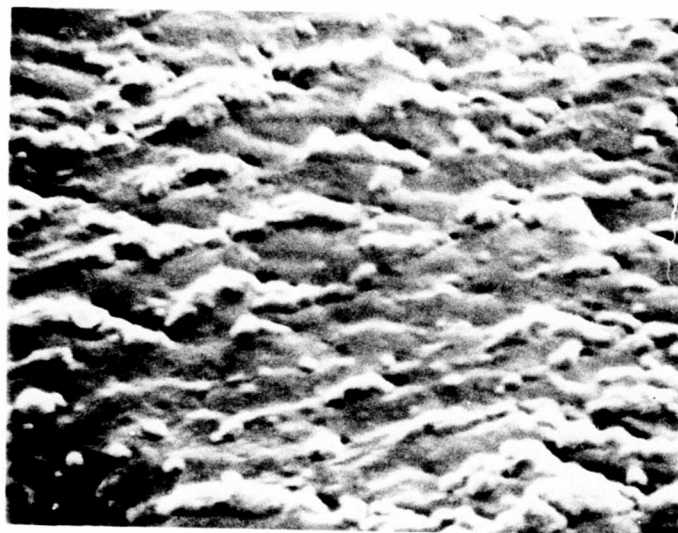
A study on the effects of anodic application potential was undertaken. Panels of 7075-T6 bare were surface treated with the standard FPL etch surface treatment and electroprimed from 20 to 60 volts at a constant application time of 20 seconds. Scanning electron microscope (SEM) photographs were taken and are shown in Figures 2 through 5. These photographs indicate a heavier deposit of oxide as the application voltage increases. This increase is also noted in thickness measurements taken from the SEM photomicrographs. Wedge test panels were then prepared at incremental potential application steps. The primer was cured at 275F for 30 minutes and bonded with FM-123-2. A summary of the test results of the initial voltage application study for the anodic polyester is presented in Table 12.

TABLE 12. EFFECTS OF APPLICATION POTENTIALS ON ANODIC POLYESTER PRIMER (SA4191)

APPLICATION POTENTIAL (VOLTS)	ELECTROPRIMER FILM THICKNESS (MIL)	AVERAGE OXIDE THICKNESS ⁽⁷⁾ (ANGSTROMS)	WEDGE TEST ⁽⁷⁾ CRACK EXTENSION (IN) 24 HOUR GROWTH
20	0.05	825	0.7
30	0.10	925	1.2
45	0.40	1100	1.6
60	0.45	1325	1.8

- NOTES:
1. Reported values are averages of 5 determinations.
 2. Exposure conditions - 120°F, 95% RH.
 3. Primer cured at 275°F for 30 minutes.
 4. 0.125 inch thick 7075-T6 bare adherends.
 5. Primer application time was 20 seconds.
 6. Specimens bonded with FM-123-2, 0.085psf film adhesive.
 7. FPL etch surface preparation.

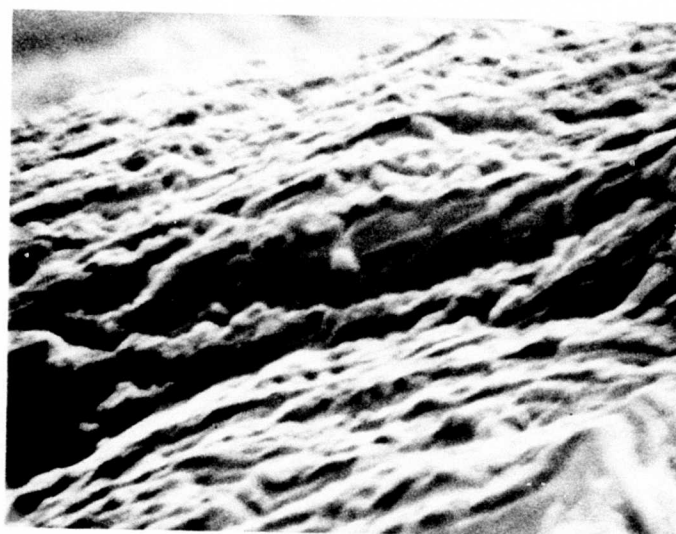
The data in Table 12 shows an increase in primer film thickness with increasing application potential and an increase in oxide thickness which was the desired result. However, wedge test performance diminished almost linearly with the increase in application voltage which is directly opposite the predicted result.



7075-T6 BARE

20,000X

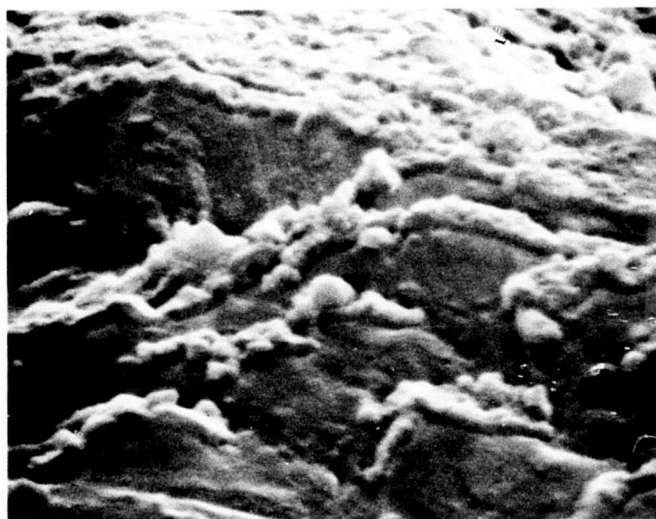
FIGURE 2. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 20 VOLTS FOR 90 SECONDS



7075-T6 BARE

20,000X

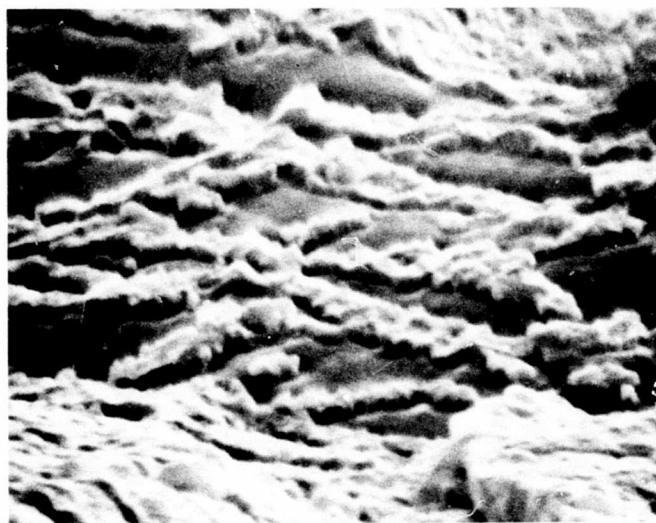
FIGURE 3. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 30 VOLTS FOR 90 SECONDS



7075-T6 BARE

20,000X

FIGURE 4. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 45 VOLTS FOR 90 SECONDS



7075-T6 BARE

20,000X

FIGURE 5. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 60 VOLTS FOR 90 SECONDS

To determine the effects of application time on the polyester primer system, a series of 7075-T6 bare panels were primed at various times from 15 to 60 seconds. The panels were all primed at a constant potential of 30 volts over an FPL etch surface treatment. SEM analyses, wedge tests and thickness determinations were conducted. Although resolution in the SEM analysis did not permit thickness readings to be taken, there was no apparent increase in the oxide thickness with increasing application times as was noted with increasing voltage application. Further, the film thickness and wedge test crack extension did not change drastically as was evidenced in the previous test. After discussion with the resin formulator it was concluded that two series of tests should be conducted. First, the primer should be applied at a maximum voltage in minimum time, and secondly minimum application voltages should be tried. It was then determined that a 70 volt, 3 second application was the maximum level which still produced a uniform film. Higher voltages produced "globular" irregular deposits with pin-holes caused by "gassing." Again wedge test panels of 7075-T6 bare were primed over an FPL surface treatment and bonded with FM-123-2 adhesive. A series of panels were electroprimed at 30 volts and the reference primer BR-127 served as a control. The test results are tabulated in Table 13, and the SEM photomicrographs are shown in Figures 6 and 7. These tests confirmed that the increase in anodic application potential deleteriously affected the bonding character of the electroprimer system. A study of lower application voltages was then undertaken. A 29 percent solids bath was prepared to increase the bath conductivity to permit application at minimum potentials. Normal bath solids content is 10 to 12 percent. A series of 7075-T6 bare coupons were prepared to determine the minimum application potential possible to provide a continuous coating. All coupons were FPL etched and electroprimed at a constant time of 30 seconds.

Application potentials of 15, 12 and 8 volts were then selected to run a series of wedge tests. Results of these tests are presented in Table 14 and Figures 8 through 10.

This test data indeed confirmed that the lower priming potentials were desirable relative to wedge test response. Based on the test results on the anodic polyester application it appeared that the anodic application was discrepant in the process scheme where, heretofore, the polyester resin itself was suspect due to the potential hydrolytic reversion of the resin. A verification test was devised to clarify this anomaly. This test consisted of electropriming wedge test panels of 7075-T6 bare over

TABLE 13. EFFECTS OF MAXIMUM APPLICATION POTENTIALS ON ANODIC POLYESTER PRIMER (SA4191)

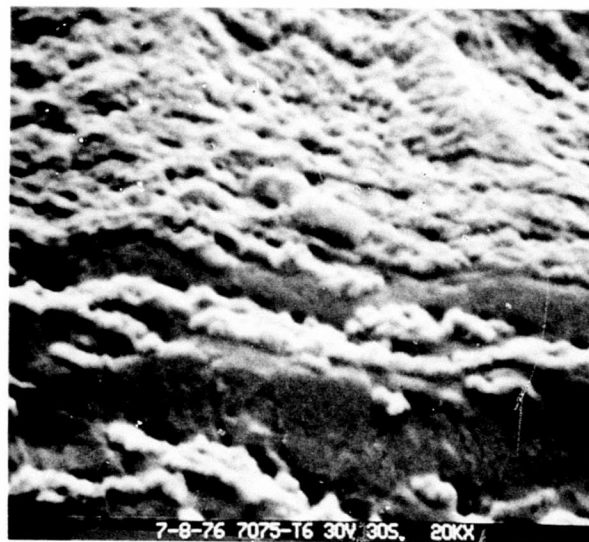
APPLICATION POTENTIAL (VOLTS)	ELECTROPRIMER FILM THICKNESS (MIL)	AVERAGE OXIDE THICKNESS ⁽⁷⁾ (ANGSTROMS)	WEDGE TEST ⁽⁷⁾ CRACK EXTENSION (IN) 24 HOUR EXPOSURE
70	0.23	3000	1.9 (4)
30	0.10	1200	0.9
BR-127 Control	0.21	Not Determined	0.1

- NOTES:
1. Reported values on the average of 5 determinations.
 2. Exposure conditions - 120°F, 95% RH.
 3. Primer cured at 275°F for 30 minutes.
 4. One hour reading - discontinued test.
 5. 7075-T6 bare aluminum.
 6. Specimens bonded with FM-123-2, 0.085psf film adhesive.
 7. FPL etch surface preparation.

TABLE 14. EFFECTS OF MINIMUM APPLICATION POTENTIAL FOR THE ANODIC POLYESTER PRIMER (SA4191)

APPLICATION POTENTIAL (VOLTS)	AVERAGE ELECTROPRIMER FILM THICKNESS (MIL)	AVERAGE OXIDE THICKNESS ⁽⁶⁾ (ANGSTROMS)	WEDGE TEST ⁽⁶⁾ CRACK EXTENSION (IN) 24 HOUR GROWTH
15	0.19	1200	1.5
12	0.15	1000	1.3
8	0.12	975	0.5

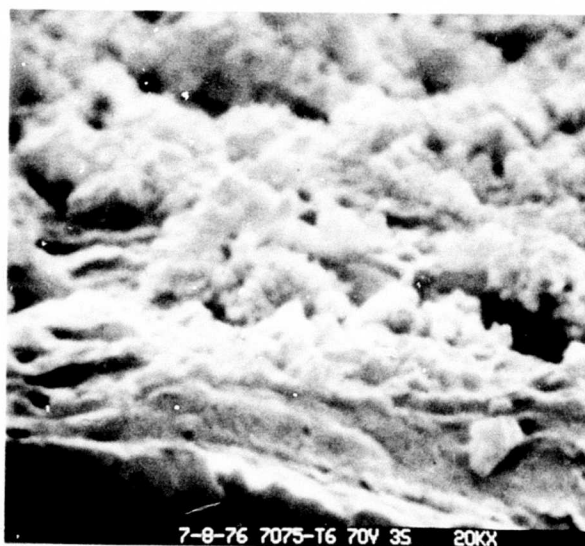
- NOTES:
1. Reported values are averages of 5 determinations.
 2. Exposure conditions - 120°F, 95% RH
 3. Primer cured at 275°F for 30 minutes.
 4. 0.125 inch thick 7075-T6 bare adherends.
 5. Specimens bonded with FM-123-2, 0.085psf film adhesive.
 6. FPL etch surface preparation.



7075-T6 BARE

20,000X

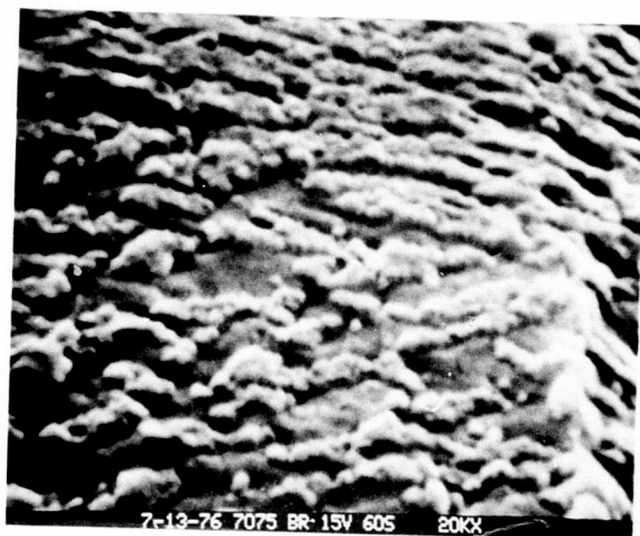
FIGURE 6. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 30 VOLTS FOR 30 SECONDS



7075-T6 BARE

20,000X

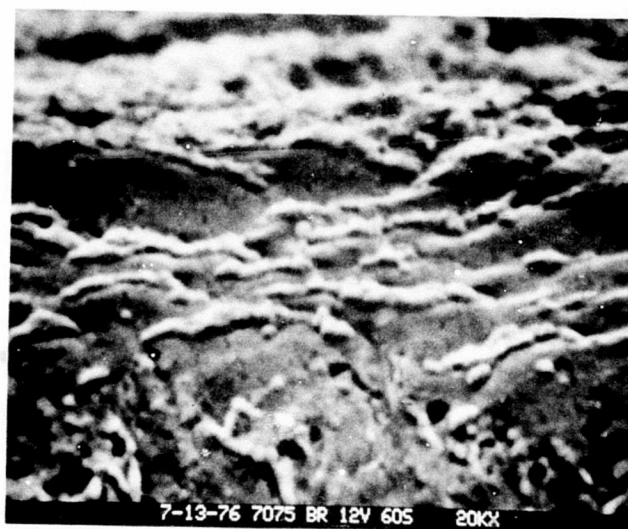
FIGURE 7. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 70 VOLTS FOR 3 SECONDS



7075-T6 BARE

20,000X

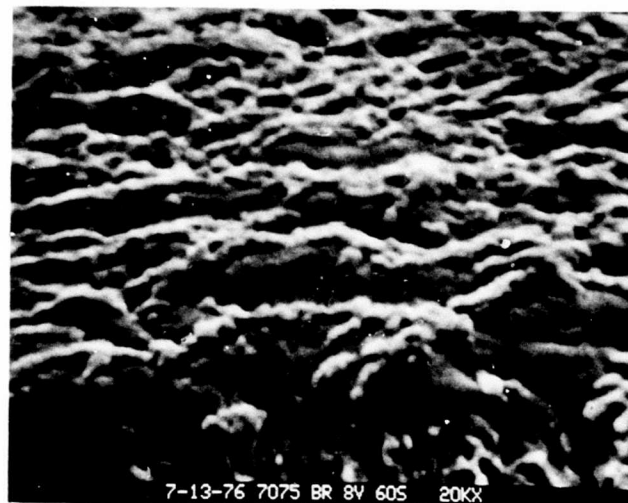
FIGURE 8. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 15 VOLTS FOR 60 SECONDS



7075-T6 BARE

20,000X

FIGURE 9. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 12 VOLTS FOR 60 SECONDS



7075-T6 BARE

20,000X

FIGURE 10. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 8 VOLTS FOR 60 SECONDS

an FPL etch surface treatment at 30, 50 and 70 volts, removing the electroprimer by ultrasonic dissolution in solvent (MEK), repriming the panels with BR-127 and subsequently bonding with FM-123-2 adhesive and then determining crack growth in the wedge test. An identical set of electroprimed and cured panels served as controls. This test data is presented in Table 15.

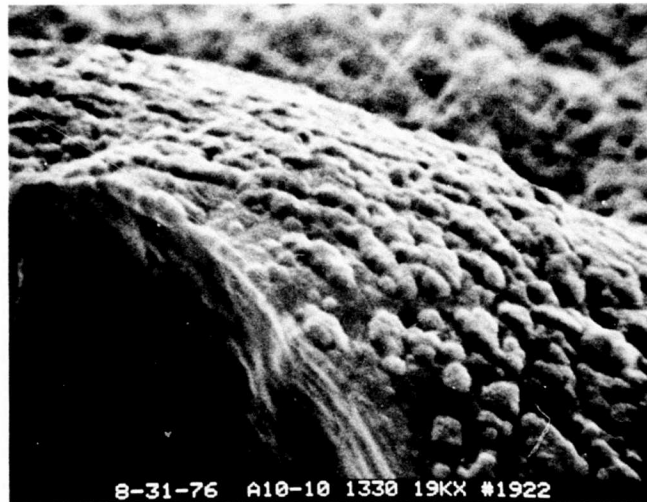
Indeed, the test data clearly indicates that anodic electrodeposition affects the surface treatment oxide as determined by this test method. Once this has occurred the primer used has a negligible effect on performance of that surface in a bonded joint. A decision was made to investigate the cathodic electropriming method of application of primers as opposed to developing the anodic method through application at low potentials from a high solids bath. Therefore, a 400F curing modified epoxy cathodic applied resin was selected for preliminary test investigations. SEM analyses coupons of bare 7075-T6 were prepared with the FPL etch surface treatment and then cathodically electroprimed with the SA 4521 primer at 10, 20, 35, 50 and 70 volts. The electroprimer was then removed by ultrasonic solvent cleaning and SEM analyses performed. The SEM analyses indicated a normal oxide pattern when compared to the FPL etch baseline. There was no evidence of "feathering" of the oxide experienced with anodic deposition at higher application potentials.

TABLE 15. STUDY OF ANODIC ELECTROPRIMER SUBSEQUENTLY REMOVED AND PRIMED WITH BR-127 ADHESIVE PRIMER

SPECIMEN IDENTIFICATION	WEDGE TEST CRACK EXTENSION (IN.) ONE-HOUR EXPOSURE
70 Volt, 3 second control ⁽¹⁾	1.4
70 Volt, 3 second + BR-127 ⁽²⁾	1.3
50 Volt, 15 second control ⁽¹⁾	1.3
50 Volt, 15 second + BR-127 ⁽²⁾	1.3
30 Volt, 30 second control ⁽¹⁾	0.6
30 Volt, 30 second + BR-127 ⁽²⁾	0.8

- NOTES:
1. Anodic electroprimer not removed.
 2. Anodic electroprimer removed, dried and then primed with BR-127 and cured at 250°F for 60 minutes.
 3. Reported values are averages of 5 determinations.
 4. Exposure conditions 120°F, 95% RH.
 5. 0.125 inch thick 7075-T6 bare adherends
 6. Specimens bonded with FM-123-2, 0.085psf film adhesive

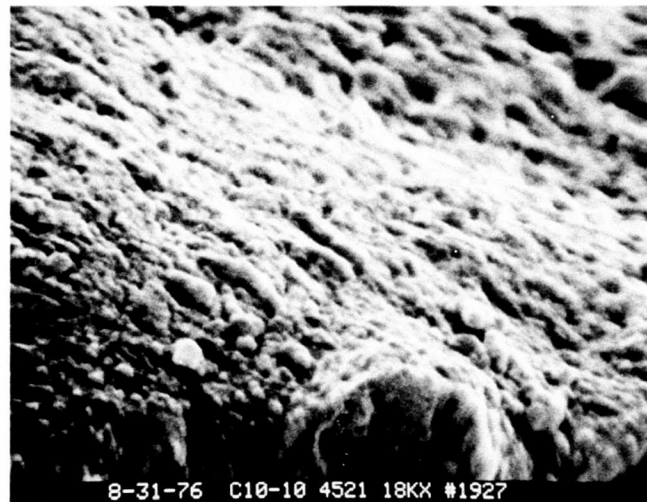
Based on these preliminary findings and excellent wedge test results, reference Table 5, comparative SEM analyses were run on the anodic versus cathodic deposition materials over a voltage range of 10 to 70 volts on the FPL surface treatment. These analyses showed thicker, bulkier oxide deposits by the anodic application process. The comparative photomicrographs of this study are shown in Figures 11 through 20. Two time and two voltage application extremes were selected for analyses of oxide thickness change relative to the cathodic application process. SEM examinations were performed on standard "hair-pin" specimens of 7075-T6 bare treated with FPL etch and then cathodically electrodeposited at different voltages under constant time and different times under constant voltage. The SEM analyses show that oxide thickness increases as a function of time. This is shown in Figures 21 and 22. The oxide thickness increased from approximately 1000Å to approximately 4000Å when increasing the application time from 10 to 100 seconds while maintaining the application potential constant at 40 volts.



7075-T6 BARE

19,000X

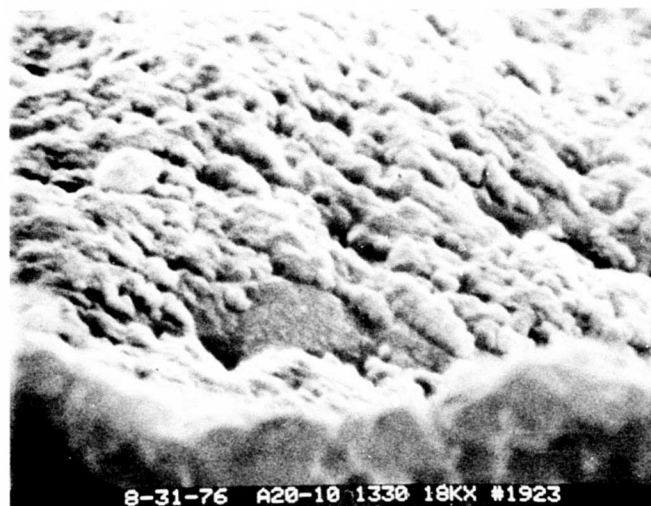
FIGURE 11. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 10 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

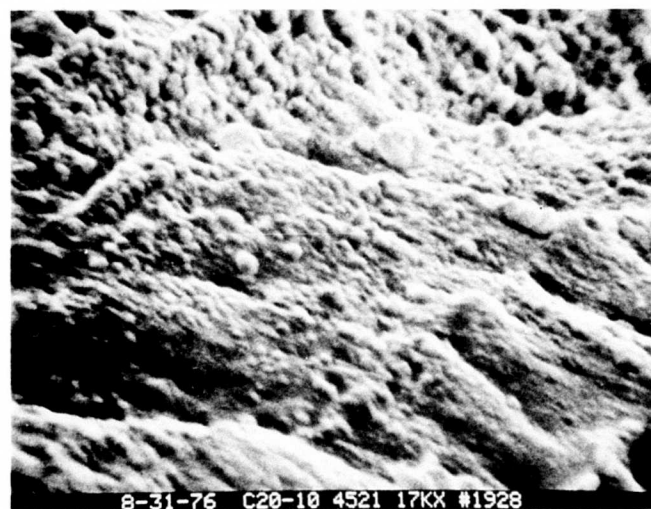
FIGURE 12. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 10 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

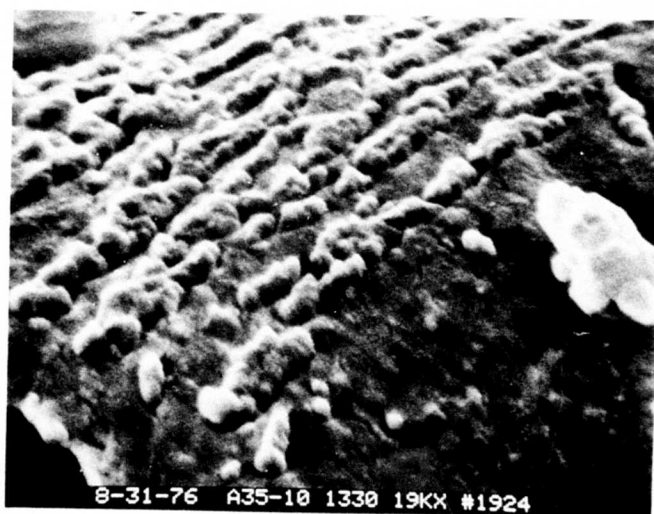
FIGURE 13. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 20 VOLTS FOR 10 SECONDS



7075-T6 BARE

17,000X

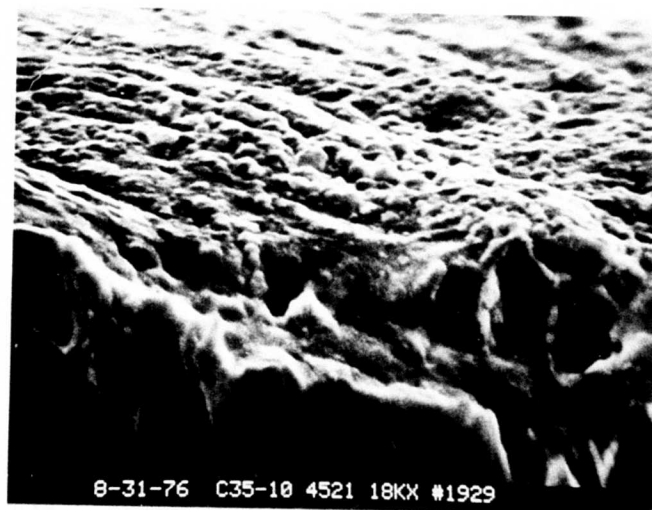
FIGURE 14. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 20 VOLTS FOR 10 SECONDS



7075-T6 BARE

19,000X

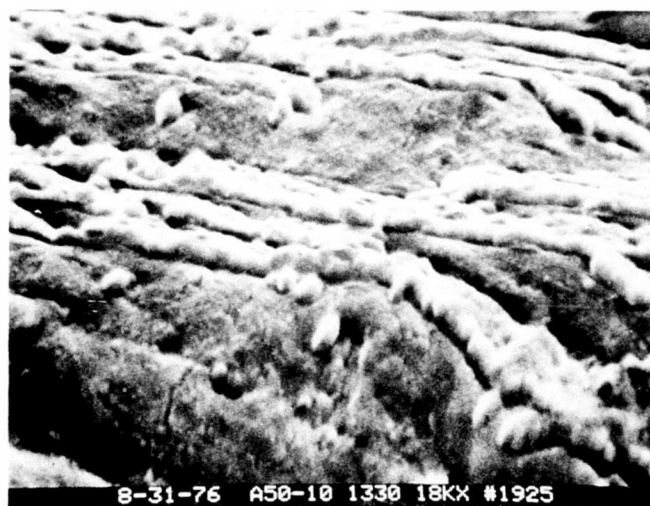
FIGURE 15. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 35 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

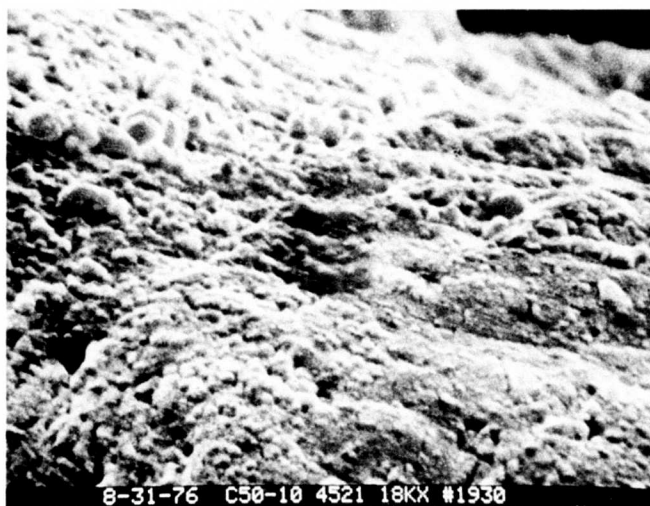
FIGURE 16. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 35 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

FIGURE 17. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 50 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

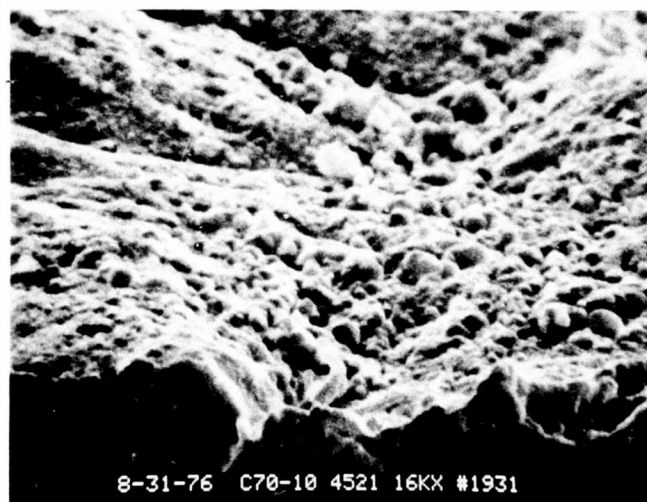
FIGURE 18. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 50 VOLTS FOR 10 SECONDS



7075-T6 BARE

18,000X

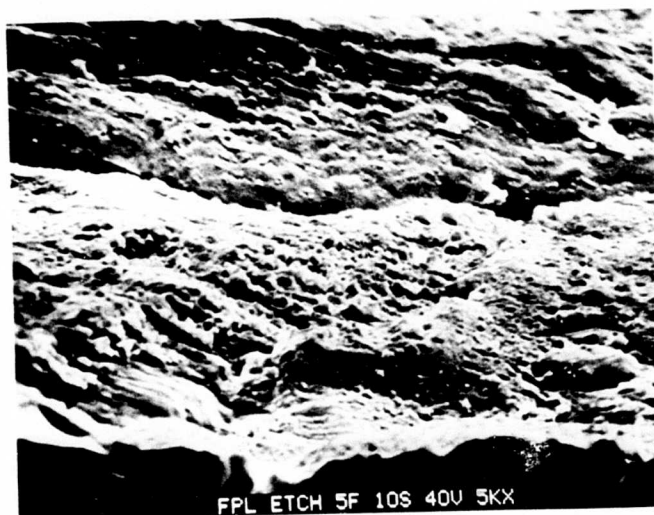
FIGURE 19. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM ANODICALLY ELECTROPRIMED
AT 70 VOLTS FOR 10 SECONDS



7075-T6 BARE

16,000X

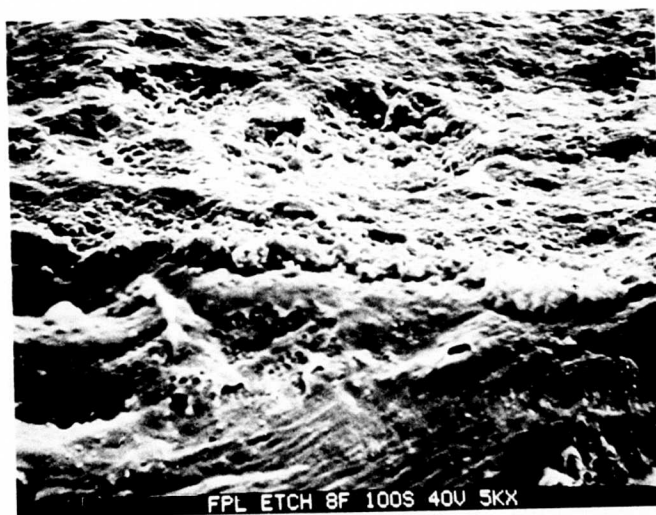
FIGURE 20. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 70 VOLTS FOR 10 SECONDS



7075-T6 BARE

5000X

FIGURE 21. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 40 VOLTS FOR 10 SECONDS



7075-T6 BARE

5000X

FIGURE 22. SEM PHOTOGRAPH - OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 40 VOLTS FOR 100 SECONDS

Similarly, specimens coated at different voltages were prepared and examined. Electrodeposition time was held constant at 20 seconds. Figures 23 and 24 show the 10 volt and 100 volt photomicrographs. The oxide thickness increased as voltage increased from approximately 1500\AA to 6000\AA in thickness. The oxide is behaving as a porous oxide, in that time and voltage both affect the oxide thickness.

An electron diffraction analyses was undertaken to determine if any change in oxide character could be established between a standard FPL etch surface treated substrate as opposed to one that had been FPL etched and subsequently cathodically electroprimed. Specimens of 7075-T6 bare were prepared which provided the proper oxide thickness of 100\AA to 400\AA . Electron diffraction analyses showed the oxide to be primarily α -boehmite in both specimens. A few weak diffraction spots were unaccounted for during analyses. These appear to match the α -corundum structure. Specimens prepared with FPL and also FPL plus cathodic electrodeposition showed the same crystalline compounds present on the surface. The existence of the additional diffraction spots had not been noted in previous work. In summary, oxide differences attributable to cathodic electropriming over the FPL treated surfaces are not discernible by electron diffraction analyses.

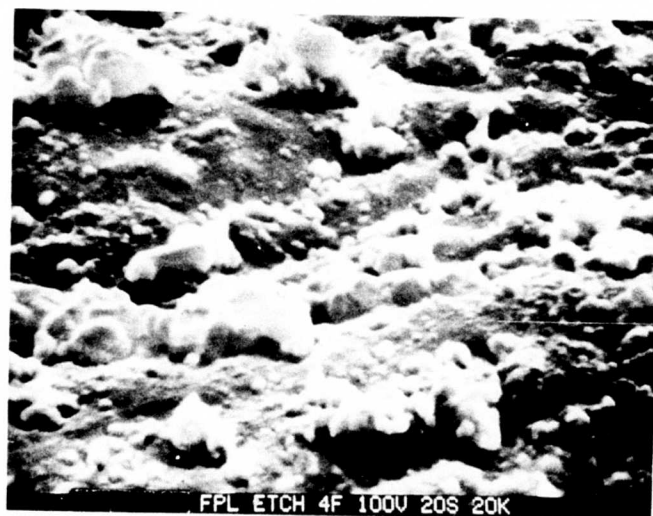
The anodic deposition predictably will increase the oxide thickness as a consequence of electrochemical oxidation of the aluminum substrate. This occurs at the initial application of the anodic potential prior to reaching the Zener potential of the electrophoretic resin, i.e., that potential required to migrate the negatively charged particle towards the anode. However, this oxidation is not anticipated for the cathodic application. Indeed, one should predict a slight dissolution of the oxide under the initial cathodic potential. It was noted that higher application potentials were required to form the proper thickness resin film with the modified epoxy resin system which did not incorporate a corrosion inhibitor. The primer development effort of Phase I showed difficulty in utilizing strontium chromate as an inhibitor. Subsequent development formulations incorporating the Nalzin SC-1 inhibitor proved to be stable at higher, i.e., 5%, concentrations and was thus incorporated. Apparently there exists a leveling in electrical stability of the primer system with the zinc phospho oxide complex which permits application at lower potentials. After final selection of the contract primer formulation which utilized a new crosslinker (catalyst) and the Nalzin SC-1 inhibitor, a series of application parameter studies utilizing the SEM were undertaken to verify the acceptability of the cathodic application process. Standard "hair-pin" specimens



7075-T6 BARE

20,000X

FIGURE 23. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 10 VOLTS FOR 20 SECONDS



7075-T6 BARE

20,000X

FIGURE 24. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE
TREATED ALUMINUM CATHODICALLY ELECTROPRIMED
AT 100 VOLTS FOR 20 SECONDS

of 7075-T6 bare pretreated with the FPL etch and also the 10 volt phosphoric acid anodize surface treatment were utilized to study the effects of application voltage, application time and application rate. Unprimed specimens of the FPL etch and 10 volt phosphoric acid anodize surface treatments served as controls. Table 16 lists the oxide thickness measurements of the test series and clearly indicates that no change in either of the two surface treatment oxides is occurring under the test conditions. Figures 25 to 42 are photomicrographs of the specimens assessing the cathodic process application variables.

TABLE 16. EFFECTS OF CATHODIC ELECTRODEPOSITION
ON FPL ETCH AND 10 VOLT PHOSPHORIC ACID ANODIZE

CATHODIC APPLICATION VARIABLE	OXIDE THICKNESS (ANGSTROMS)	
	SURFACE TREATMENT	
	10 VOLT ANODIZE	FPL ETCH
APPLICATION POTENTIAL ⁽¹⁾		
0 - volt (control)	5600	1100
25 - volts	5900	1110
30 - volts	5800	1110
35 - volts	5750	1110
APPLICATION TIME ⁽²⁾		
0 - seconds (control)	5600	1375
5 - seconds	5500	1375
10 - seconds	5120	1375
30 - seconds	5625	1375
APPLICATION RATE ⁽³⁾		
0 - seconds (control)	5600	1100
3 - seconds	5600	1100
5 - seconds	5200	1250
10 - seconds	5175	1275

- NOTES: 1. 15 second application time.
2. 30 volt application voltage.
3. Time to reach application potential of 30 volts linearly.
4. 0.063 inch thick 7075-T6 bare aluminum alloy

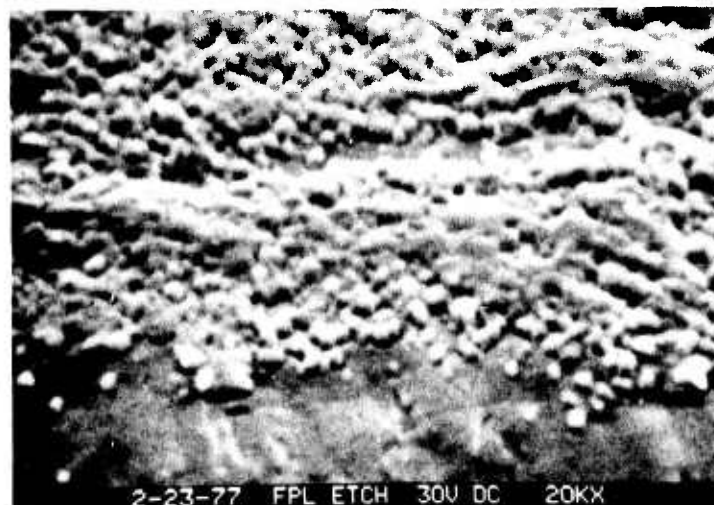
FIGURE 25. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED AT 25 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

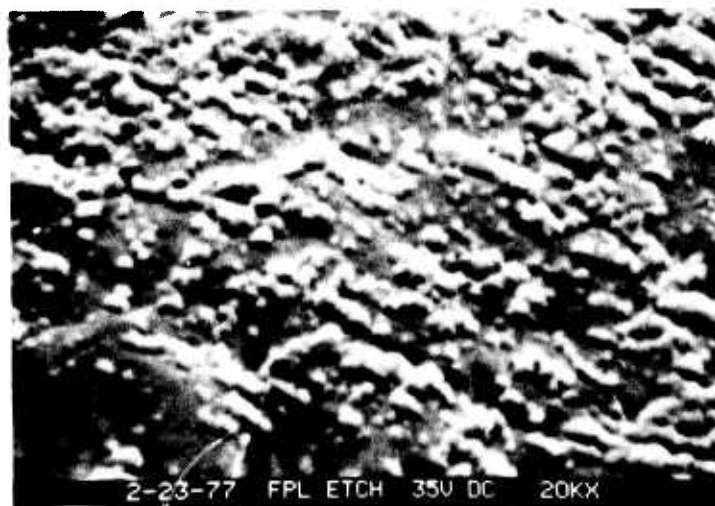
FIGURE 26. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED AT 30 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

FIGURE 27. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED AT 35 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

FIGURE 28. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 25 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

FIGURE 29. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 30 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

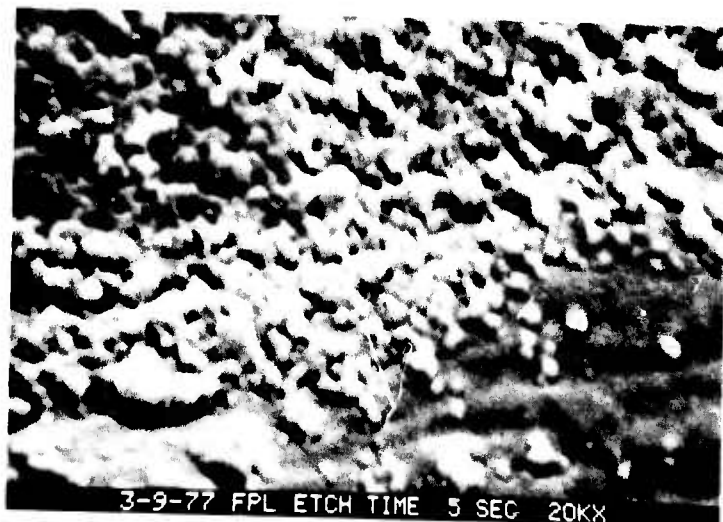
FIGURE 30. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 35 VOLTS FOR 15 SECONDS



7075-T6 BARE

20,000X

FIGURE 31. SEM PHOTO-
GRAPH — OXIDE LAYERS
ON FPL SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED AT
30 VOLTS FOR 5 SECONDS



7075-T6 BARE

20,000X

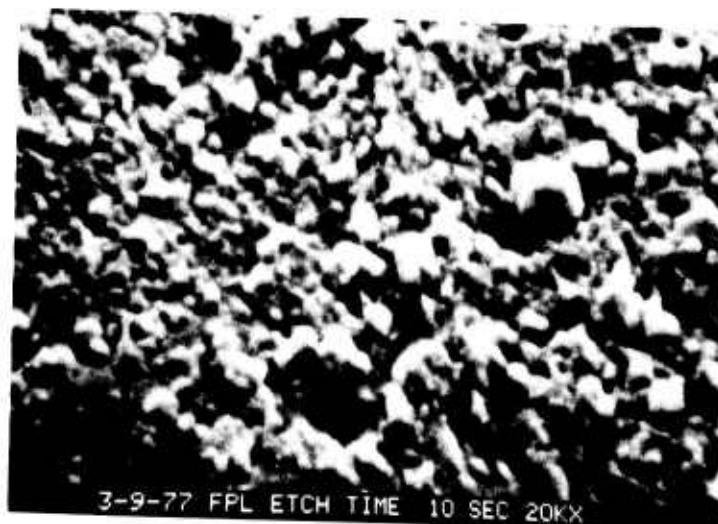
FIGURE 32. SEM PHOTO-
GRAPH — OXIDE LAYERS
ON FPL SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED AT
30 VOLTS FOR 10 SECONDS



7075-T6 BARE

20,000X

FIGURE 33. SEM PHOTO-
GRAPH — OXIDE LAYERS
ON FPL SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED AT
30 VOLTS FOR 30 SECONDS



7075-T6 BARE

20,000X

FIGURE 34. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 30 VOLTS FOR 5 SECONDS



7075-T6 BARE

20,000X

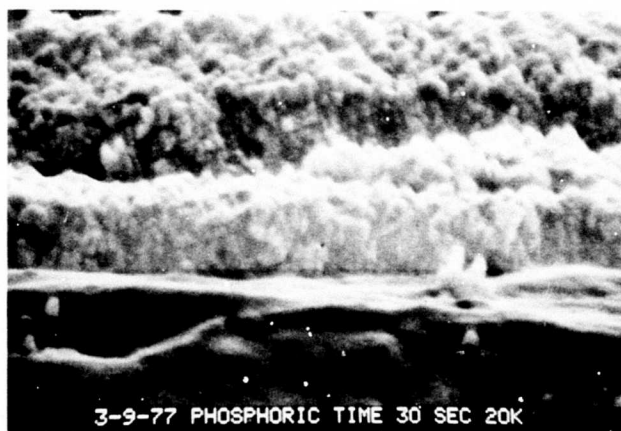
FIGURE 35. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 30 VOLTS FOR 10 SECONDS



7075-T6 BARE

20,000X

FIGURE 36. SEM PHOTOGRAPH — OXIDE LAYERS ON 10 VOLT PHOSPHORIC ACID ANODIZE SURFACE TREATED ALUMINUM CATHODICALLY ELECTRO-PRIMED AT 30 VOLTS FOR 30 SECONDS



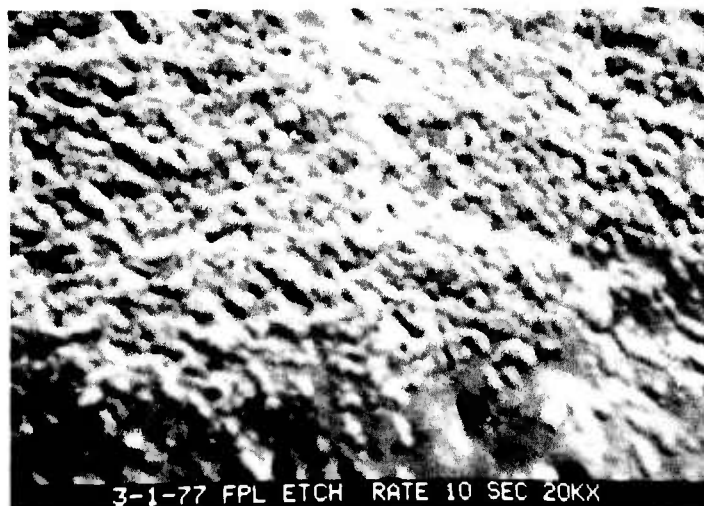
7075-T6 BARE

20,000X

FIGURE 37. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED

POTENTIAL APPLICATION RATE 3 VOLTS/SECOND TO REACH 30 VOLTS

NOTE: TIME TO REACH 30 VOLTS IS 10 SECONDS



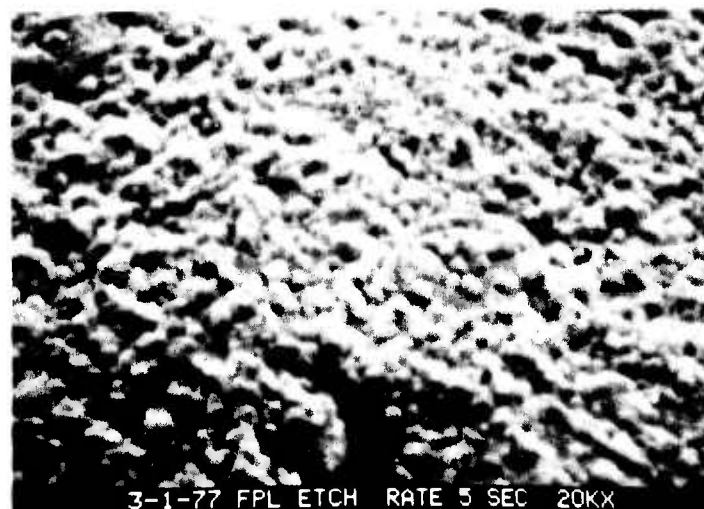
7075-T6 BARE

20,000X

FIGURE 38. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED

POTENTIAL APPLICATION RATE 6 VOLTS/SECOND TO REACH 30 VOLTS

NOTE: TIME TO REACH 30 VOLTS IS 5 SECONDS



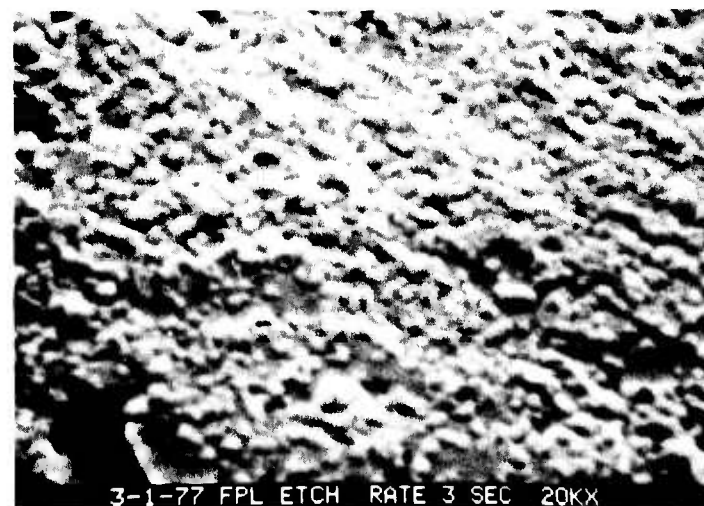
7075-T6 BARE

20,000X

FIGURE 39. SEM PHOTOGRAPH — OXIDE LAYERS ON FPL SURFACE TREATED ALUMINUM CATHODICALLY ELECTROPRIMED

POTENTIAL APPLICATION RATE 10 VOLTS/SECOND TO REACH 30 VOLTS

NOTE: TIME TO REACH 30 VOLTS IS 3 SECONDS



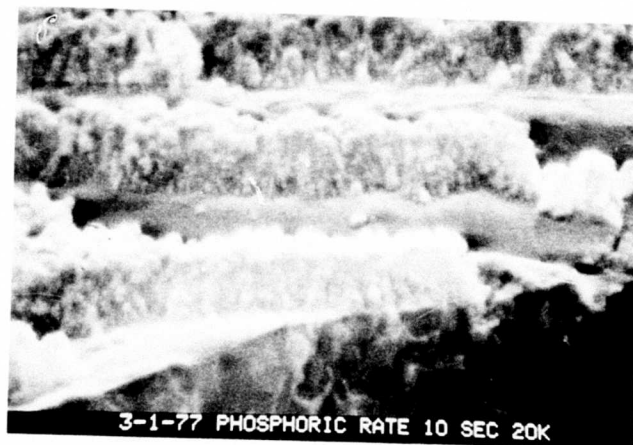
7075-T6 BARE

20,000X

FIGURE 40. SEM PHOTO-
GRAPH — OXIDE LAYERS ON
10 VOLT PHOSPHORIC ACID
ANODIZE SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED

POTENTIAL APPLICATION
RATE 3 VOLTS/SECOND TO
REACH 30 VOLTS

NOTE: TIME TO REACH
30 VOLTS IS 10 SECONDS



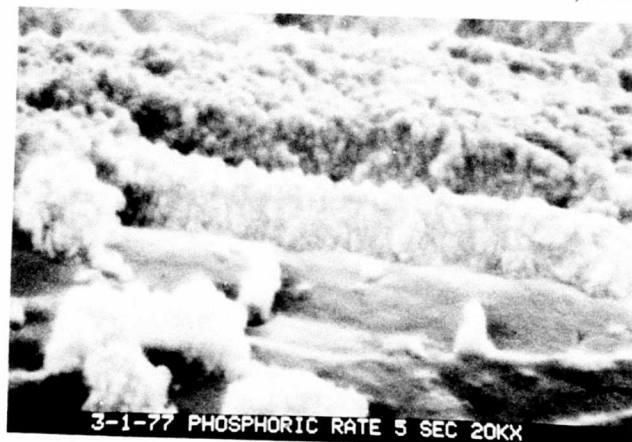
7075-T6 BARE

20,000X

FIGURE 41. SEM PHOTO-
GRAPH — OXIDE LAYERS ON
10 VOLT PHOSPHORIC ACID
ANODIZE SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED

POTENTIAL APPLICATION
RATE 6 VOLTS/SECOND TO
REACH 30 VOLTS

NOTE: TIME TO REACH
30 VOLTS IS 5 SECONDS



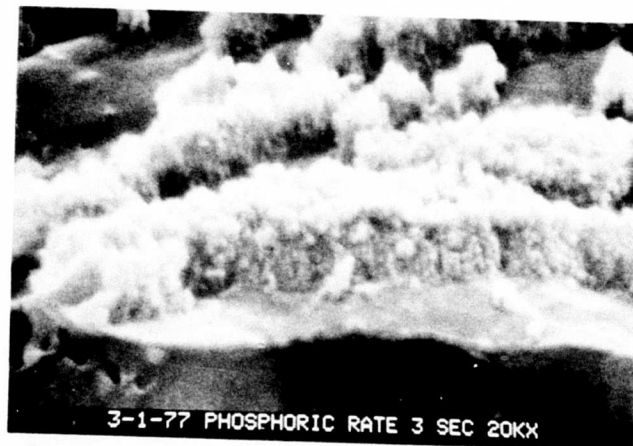
7075-T6 BARE

20,000X

FIGURE 42. SEM PHOTO-
GRAPH — OXIDE LAYERS ON
10 VOLT PHOSPHORIC ACID
ANODIZE SURFACE TREATED
ALUMINUM CATHODICALLY
ELECTROPRIMED

POTENTIAL APPLICATION
RATE 10 VOLTS/SECOND TO
REACH 30 VOLTS

NOTE: TIME TO REACH
30 VOLTS IS 3 SECONDS



7075-T6 BARE

20,000X

In summary, the process application study was initiated with the anodic application process. This study indicated that anodic electropriming requires extremely low voltage deposition from a high solids bath to minimize oxide interface effects. The study then investigated the cathodic deposition process. Although initial findings indicated an oxide thickness growth, electron diffraction analyses showed the crystallographic structure of the cathodically primed substrate identical to the unprimed control. SEM analyses of the application process variables utilizing the selected contract formulation indicates no change in appearance or thickness of the surface preparation oxide when electropriming over an FPL etch or 10 volt phosphoric acid surface treatment. Based on the findings of this development phase, together with information evolved in Phase I, the application parameters for applying the modified epoxy electroprimer were defined as follows:

Cathodic Voltage ⁽¹⁾	— 30 ± 3 Volts
Current Density ⁽²⁾	— 12 APSF
Time ⁽³⁾	— 15-30 Seconds
Cure Temperature	— 345°F
Cure Time	— 30 Minutes

- (1) The application voltage is preset to 30 volts.
- (2) The current control is increased from zero until a 30 volt potential is reached. This is done in about five seconds. The current "spikes" to about 12 APSF and decays to zero in 1-2 seconds.
- (3) The 30 volt cathodic potential is held for an additional 15 seconds for deposited film densification.

These process parameters provide a continuous uniform primer film 0.18 ± 0.02 mil in thickness and were utilized to electroprime the required test aluminum substrates for Phase III evaluations.

PHASE III — PRIMER PERFORMANCE ASSESSMENT

Phase I of this development program studied the polyester, acrylic, and epoxy electroprimers as candidates in the exploratory development of a corrosion inhibiting primer. Phase II investigated the anodic and the cathodic electropriming processes, and Phase III was a comparative performance evaluation of the C-5301 electroprimer and application process to the current state-of-the art BR-127 adhesive bonding primer.

Task 1 — Paint Systems Compatability

This task evaluated the selected contract primer, C-5301, for flexibility, corrosion resistance, and adhesion, alone and in combination with the BR-127 adhesive primer, the MIL-P-23377 primer, and the aliphatic urethane topcoat MIL-C-83286. The specific tests performed and the test results are presented in Table 17. It was demonstrated that the cathodically applied modified epoxy electroprimer was compatible with the state-of-the-art hand-sprayed primers and performed equally as well in the specific tests as follows:

Adhesion- The wet tape adhesion test FTMS 141, Method 6301.1, was performed on bare 2024-T3 and 7075-T6 aluminum alloys. Normally, the surface preparation of aluminum alloy utilizes a room temperature deoxidizer followed by a chromate conversion coating to promote adhesion and inhibit corrosion. The surface preparation for this test was the FPL etch only and there were no observations of any adhesion problems. Indeed, no lack of adhesion has been noted with electroprime systems by Northrop during its last five years of study on electroprimers.

Flexibility Test — The flexibility test conducted have shown that the electroprimer films are extremely flexible. This is attributable to the film thickness normally 0.1 to 0.4 mil. In the flexibility test, the panels are bent over a conical mandrel. There was no evidence of cracking, peeling, or incompatibility noted using the contract primer C-5301 combined with the MIL-P-23377 primer and the aliphatic urethane topcoat, MIL-C-83286. To further evaluate the electroprimer system compatibility and flexibility, test panels of the C-5301 primer, MIL-P-23377 primer, and topcoat were impact tested with a Gardner Laboratory Heavy-Duty Impact Tester. The test panels were impacted at 80, 120, and 160-in/lbs. There was no observed cracking or chipping with any of the finish systems even when the aluminum substrate failed at 160-in/lbs.

Corrosion Resistance Test — The corrosion resistance of the C-5301 primer was evaluated over the FPL etch surface treatment on 2024-T3 bare and 7075-T6 bare aluminum alloys. This primer was acceptable alone and in combination with the referenced paint primers and topcoats and was comparable with the BR-127 control primer. A few minute "pin-holes" were noted in the electroprimer film after 800 hours exposure, but corrosion did not migrate at the "pin-hole" sites. Results of the 1000-hour exposure salt spray tests were acceptable. The "pin-hole" effect was also noted

TABLE 17. PAINT SYSTEMS COMPATIBILITY TESTS-SPECIMEN PREPARATION AND TEST RESULTS

TEST	FPMs No. 141 Method	TEST ALLON		SURFACE TREATMENT FPL ETCH	PRIMER			TOPCOAT MIL-C-83286	NO. OF SPECIMENS	COMMENTS	RESULTS	
		7053-T6 BARE	2021-T3 BARE		BR-127	ELECTRO PRIMER	MIL-P-23377					
Adhesion (Wet Tape Test)	6301, 1 ⁽¹⁾	N		N		N		N	3		No observed failures of primers and primer topcoat combinations. Paint systems remain intact on test panels.	
		N		N	N	N		N	3			
			N	N		N		N	3			
			N	N		N		N	3			
Flexibility	6222(1)		N	N		N		N	3		There was no evidence of chipping, cracking or flaking of coatings when bent over a conical mandrel.	
			N	N		N		N	3			
Corrosion Resistance	N	N		N					3	BR-127 only	No visible corrosion after 1000 hours exposure	
		N		N	N				3			
		N		N	N	N		N	3	BR-127 with MIL-P-23377 Primer	No visible corrosion after 1000 hours exposure	
		N		N	N	N		N	3			
	N	N		N	N				3	BR-127 with 23377 primer + MIL-C-83286 topcoat	No visible corrosion after 1000 hours exposure	
		N		N	N		N		N	3	BR-127 with MIL-C-83286 topcoat	No visible corrosion after 1000 hours exposure
		N		N	N					3		
Salt Spray (5% Fog) 95°F	6061 (1)	N		N				N	3	EDOC* only	No visible corrosion after 1000 hours exposure	
		N		N	N					3		
		N		N	N	N		N	3	EDOC with MIL-P-23377 primer	No visible corrosion after 1000 hours exposure	
		N		N	N					3	EDOC with 23377 primer + MIL-C-83286 topcoat	No visible corrosion after 1000 hours exposure
		N		N	N					3	EDOC with MIL-C-83286 topcoat	No visible corrosion after 1000 hours exposure

•EDOC Electrodeposited organic coating

NOTE: (1) Test method procedure included in Appendix B

in previous Northrop studies utilizing the anodic polyester system. At that time, it was determined that a conventional chromate conversion coat prior to electropriming protected the aluminum substrate for over 5000 hours of salt fog exposure without any significant signs of corrosion. An additional series of 2024-T3 bare and 7075-T6 bare aluminum panels were prepared for corrosion resistance testing. The current state-of-the-art surface preparation was used before priming and included a chromate conversion coating. After 2500 hours exposure to salt fog there was no sign of corrosion, pitting or loss of surface gloss when examined at 20X magnification.

Results of the salt fog corrosion test are presented pictorially in Figures 43 to 46.

Task 2 — Adhesive Bonding Characteristics

This task was conducted to assess the electroprimer and application process developed in Phase I and Phase II of this program. In this assessment, the 10 volt phosphoric acid anodize and the FPL etch surface treatments were utilized prior to electropriming and compared to the current state-of-the-art hand-sprayed adhesive primer BR-127. The assessment was performed using the FM-73, 250F curing film adhesive, and the FM-400, 350F curing film adhesive. The complete test schedule is defined in Table 18.

The test specimen configurations used are presented in the Appendix D. Results of the required tests are presented in Tables 19 through 28.

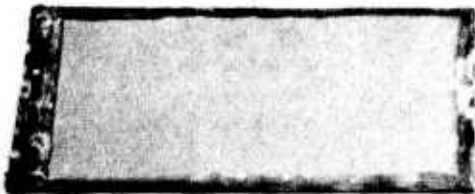
Tensile Shear Test — Tensile shear tests with the FM-73 film adhesive produced higher strengths with the C-5301 primer at ambient and 180F test temperatures and over both surface treatments compared to the BR-127 control primer. However, at the -67F test temperature, the BR-127 generally produced higher tensile shear strengths than the C-5301. However, two of the five C-5301 tensile test specimens over the 10 volt phosphoric acid anodize surface treatment produced strengths equivalent to the BR-127 primed specimens at -67F. These two specimens had combination failure modes, reference Figure 47. Typical failure modes of failed tensile shear specimen are presented pictorially in Figures 48 to 55.

The tensile shear specimens exposed to 30-day salt fog prior to testing provided very encouraging results. These tests produced results equivalent to unexposed specimens in the room temperature and only a 20% reduction was noted in the 180F temperature tests. Based on previous experience with corrosion inhibiting adhesive primers

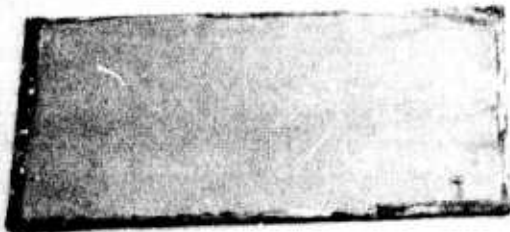
1000 HOURS
SALT SPRAY



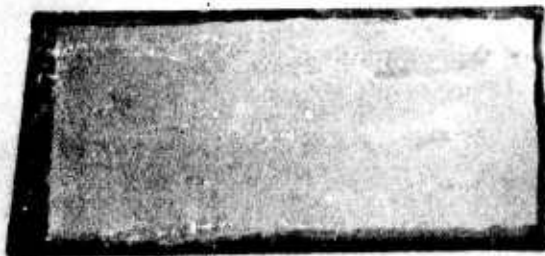
BR-127
CONTROL



C-5301
+
MIL-P-23377



BR-127
+
MIL-P-23377



C-5301 PRIMER



7075-T6 BARE

FIGURE 43. COMPATIBILITY TEST PANELS OF PRIMER SYSTEMS
AFTER 1000 HOURS SALT SPRAY — 7075-T6 BARE

1000 HOURS
SALT SPRAY

BR-127
CONTROL

C-5301
+
MIL-P-23377

BR-127
+
MIL-P-23377

C-5301 PRIMER

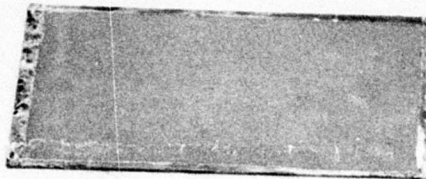
2024-T3 BARE

FIGURE 44. COMPATIBILITY TEST PANELS OF PRIMER SYSTEMS
AFTER 1000 HOURS SALT SPRAY — 2024-T3 BARE

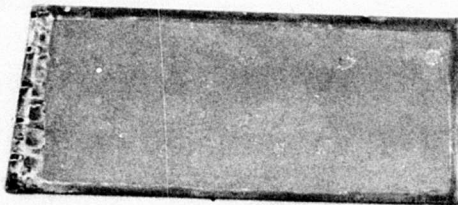
1000 HOURS
SALT SPRAY



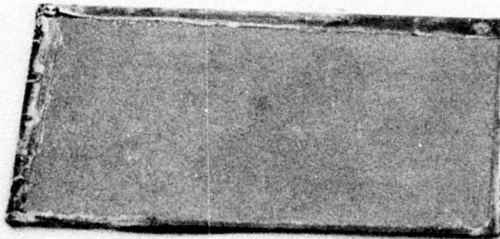
BR-127
+
MIL-P-23377
+
MIL-C-83286



BR-127
+
MIL-C-83286



C-5301
+
MIL-P-23377
+
MIL-C-83286



C-5301
+
MIL-C-83286

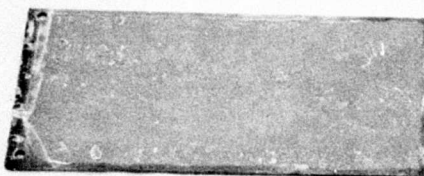
7075-T6 BARE

FIGURE 45. COMPATIBILITY TEST PANELS OF PRIMER/TOPCOAT
SYSTEMS AFTER 1000 HOURS SALT SPRAY — 7075-T6 BARE

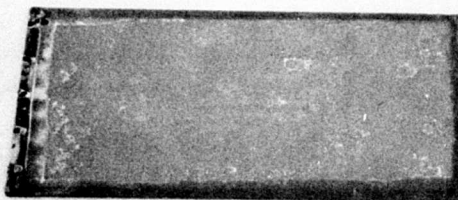
1000 HOURS
SALT SPRAY



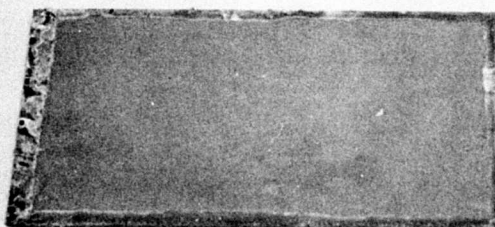
BR-127
+
MIL-P-23377
+
MIL-C-83286



BR-127
+
MIL-C-83286



C-5301
+
MIL-P-23377
+
MIL-C-83286



C-5301
+
MIL-C-83286

2024-T3 BARE

FIGURE 46. COMPATIBILITY TEST PANELS OF PRIMER/TOPCOAT SYSTEMS AFTER 1000 HOURS SALT SPRAY — 2024-T3 BARE

TABLE 18. FINAL ADHESIVE BONDING ASSESSMENT TEST SCHEDULE

TEST	TEST SEQUENCE NUMBER	SPECIMEN CONFIGURATION	TEST		SURFACE TREATMENT		PRIMER		ADHESIVE			TEST TEMPERATURE (°F)					ENVIRONMENTAL EXPOSURE	NUMBER OF SPECIMENS
			ADHEREND ALLOY 2024-T3 BARE	7075-T6 BARE	FPL ETCH	10% PHOSPHORIC ACID ANODIZE	ED/C (2)	BR-127	BR-400	FM-123-2	FM-73	FM-400	-67	AMB	180	250		
Tensile	1 (Control)	Thick Adherend		X	X							X	X ⁽¹⁾				5% Salt Solution	25
	2 (Control)	Machined		X	X		X			X		X	X ⁽¹⁾				5% Salt Solution	15
	3 (Control)	Notch		X	X	X	X			X		X	X ⁽¹⁾				5% Salt Solution	15
	4 (Control)	Tensile See Appendix D		X	X	X	X			X		X	X ⁽¹⁾				5% Salt Solution	35
Shear	1 (Control)			X	X							X	X ⁽¹⁾				5% Salt Solution	16
	2 (Control)			X	X		X			X		X	X ⁽¹⁾				5% Salt Solution	35
	3 (Control)			X	X		X			X		X	X ⁽¹⁾				5% Salt Solution	16
	4 (Control)			X	X	X	X			X		X	X ⁽¹⁾				5% Salt Solution	35
Stress	1 (Control)	Thick Adherend		X	X												100% R.H. 120°F	3
	2 (Control)	Machined		X	X			X		X		X					100% R.H. 120°F	3
	3 (Control)	Notch		X	X	X	X			X		X					100% R.H. 120°F	3
	4 (Control)	Tensile See Appendix D		X	X	X	X			X		X					100% R.H. 140°F	3
Rupture	1 (Control)			X	X												100% R.H. 140°F	3
	2 (Control)			X	X												100% R.H. 140°F	3
	3 (Control)			X	X												100% R.H. 140°F	3
	4 (Control)			X	X	X	X			X		X					100% R.H. 140°F	3
Metal To Metal Peel	1 (Control)	T-Peel See Appendix D	X	X	X							X					None	10
	2 (Control)		X	X	X							X					None	10
	3 (Control)		X	X	X							X					None	10
	4 (Control)		X	X	X							X					None	10
Metal Peel	5 (Control)		X	X	X												None	10
	6 (Control)		X	X	X												None	10
	7 (Control)		X	X	X												None	10
	8 (Control)		X	X	X												None	10

(1) Indicates Salt Spray exposure before tensile test.
(2) EDOC - Electrodeposited Organic Coating

TABLE 19. TENSILE SHEAR TEST RESULTS FOR FM-73 FILM ADHESIVE --
10V PHOSPHORIC ACID ANODIZE -- 7075-T6 BARE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-127
		ULTIMATE ⁽⁴⁾ STRENGTH (PSI)	ULTIMATE ⁽⁴⁾ STRENGTH (PSI)
FM-73-1 ⁽²⁾	-67	3650 ⁽³⁾⁽⁵⁾	
FM-73-2	-67	8450 ⁽³⁾	
FM-73-3	-67	8160	8400
FM-73-4	-67	4080 ⁽⁵⁾	8180
FM-73-5	-67	<u>4730⁽⁵⁾</u>	<u>8380</u>
Average		5810	8320
FM-73-1	Ambient	5530	
FM-73-2	Ambient	5670	
FM-73-3	Ambient	5280	5420
FM-73-4	Ambient	5460	5120
FM-73-5	Ambient	<u>5730</u>	<u>5320</u>
Average		5540	5290
FM-73-1	180	5110	
FM-73-2	180	5140	
FM-73-3	180	5180	4850
FM-73-4	180	5330	4600
FM-73-5	180	<u>5000</u>	<u>4500</u>
Average		5150	4650

(1) Average bondline thickness was 0.005 inch.

(2) C-5301 primer thickness was 0.0002 inch.

(3) Fracture analyses were performed on failed specimens

(4) All failures were cohesive except as noted.

(5) Adhesive failure.

TABLE 20. TENSILE SHEAR TEST RESULTS FOR FM-73 FILM ADHESIVE -
10V PHOSPHORIC ACID ANODIZE 7075-T6 BARE AFTER 30 DAY SALT FOG
EXPOSURE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-127
		ULTIMATE ⁽³⁾ STRENGTH (PSI)	ULTIMATE ⁽³⁾ STRENGTH (PSI)
FM-73-SS-1 ⁽²⁾	Ambient	5590	
FM-73-SS-2	Ambient	5480	
FM-73-SS-3	Ambient	5520	
FM-73-SS-4	Ambient	5760	5390
FM-73-SS-5	Ambient	<u>5670</u>	<u>5000</u>
Average		5600	5190
FM-73-SS-1	180	4350	
FM-73-SS-2	180	4220	
FM-73-SS-3	180	4650	
FM-73-SS-4	180	3700	4690
FM-73-SS-5	180	<u>4620</u>	<u>5310</u>
Average		4310	5000

NOTES: (1) Average bondline thickness was 0.005 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) All failures were cohesive.

TABLE 21. TENSILE SHEAR TEST RESULTS FOR FM-73 FILM ADHESIVE -
FPL ETCH 7075-T6 BARE

SPECIMEN (1) NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-127
		ULTIMATE (4) STRENGTH (PSI)	ULTIMATE (4) STRENGTH (PSI)
FM-73-1 (2)	-67	4220 (3)	
FM-73-2	-67	5350 (3)	
FM-73-3	-67	5720	8220
FM-73-4	-67	2940	8000
FM-73-5	-67	<u>4440</u>	<u>8200</u>
Average		4530 (5)	8140
FM-73-1	Ambient	6060	
FM-73-2	Ambient	5660	
FM-73-3	Ambient	5560	5500
FM-73-4	Ambient	5510	5280
FM-73-5	Ambient	<u>5600</u>	<u>5500</u>
Average		5680	5430
FM-73-1	180	4600	
FM-73-2	180	4600	
FM-73-3	180	4810	4850
FM-73-4	180	4890	4740
FM-73-5	180	<u>4780</u>	<u>4460</u>
Average		4750	4680

- NOTES: (1) Average bondline thickness was 0.005 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Fracture analyses performed on failed specimens.
(4) All failures were cohesive except as noted.
(5) Adhesive failures.

TABLE 22. TENSILE SHEAR TEST RESULTS FOR FM-73 FILM ADHESIVE -
FPL ETCH 7075-T6 BARE AFTER 30 DAY SALT SPRAY EXPOSURE

SPECIMEN (1) NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-127
		ULTIMATE (3) STRENGTH (PSI)	ULTIMATE (3) STRENGTH (PSI)
FM-73-SS-1 (2)	Ambient	5480	
FM-73-SS-2	Ambient	5550	
FM-73-SS-3	Ambient	5340	
FM-73-SS-4	Ambient	5520	5500
FM-73-SS-5	Ambient	<u>5420</u>	<u>5210</u>
Average		5460	5360
FM-73-SS-1	180	3710	
FM-73-SS-2	180	3790	
FM-73-SS-3	180	3440	
FM-73-SS-4	180	3660	4480
FM-73-SS-5	180	<u>3370</u>	<u>4640</u>
Average		3590	4560

NOTES: (1) Average bondline thickness was 0.005 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) All failures were cohesive.

TABLE 23. TENSILE SHEAR TEST RESULTS FOR FM-400 FILM ADHESIVE —
10V PHOSPHORIC ACID ANODIZE — 7075-T6 BARE

SPECIMEN (1) NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-400
		ULTIMATE STRENGTH (PSI)	ULTIMATE STRENGTH (PSI)
FM-400-1 (2)	-67	4860	
FM-400-2	-67	4840	
FM-400-3	-67	4980	7520
FM-400-4	-67	4700	6880
FM-400-5	-67	<u>4930</u>	<u>7490</u>
Average		4860 (4)	7300 (4)
FM-400-1	Ambient	5510	
FM-400-2	Ambient	4270	
FM-400-3	Ambient	4290	7900
FM-400-4	Ambient	4060	7460
FM-400-5	Ambient	<u>4890</u>	<u>7900</u>
Average		4600 (4)	7750 (4)
FM-400-1	180	5980	
FM-400-2	180	5980	
FM-400-3	180	5960	(3)
FM-400-4	180	6000	
FM-400-5	180	<u>5900</u>	
Average		5960 (5)	
FM-400-1	250	4350	
FM-400-2	250	4570	
FM-400-3	250	5000	5520
FM-400-4	250	4890	5450
FM-400-5	250	<u>5320</u>	<u>5500</u>
Average		4830 (5)	5490 (5)
FM-400-1	350	3510	
FM-400-2	350	3300	
FM-400-3	350	3410	4200
FM-400-4	350	3610	4390
FM-400-5	350	<u>3560</u>	<u>4320</u>
Average		3480 (5)	4300 (5)

- NOTES: (1) Average bondline thickness was 0.007 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) 180F tensile test on control specimens was not required.
(4) Adhesive failures.
(5) Combination adhesive/cohesive failures.

TABLE 24. TENSILE SHEAR TEST RESULTS FOR FM-400 FILM ADHESIVE —
10V PHOSPHORIC ACID ANODIZE 7075-T6 BARE AFTER 30 DAY SALT
FOG EXPOSURE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301 ⁽²⁾	BR-400
		ULTIMATE STRENGTH (PSI)	ULTIMATE STRENGTH (PSI)
FM-400-1 ⁽²⁾	Ambient	5730	7100 <u>6900</u> 7000 ⁽³⁾
FM-400-2	Ambient	4950	
FM-400-3	Ambient	4740	
FM-400-4	Ambient	6090	
FM-400-5	Ambient	<u>5210</u>	
Average		5340 ⁽³⁾	
FM-400-1	350	1480	3880 <u>3360</u> 3620 ⁽³⁾
FM-400-2	350	1610	
FM-400-3	350	1960	
FM-400-4	350	1770	
FM-400-5	350	<u>1690</u>	
Average		1700 ⁽³⁾	

- NOTES: (1) Average bondline thickness was 0.007 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Combination adhesive/cohesive failure.

TABLE 25. TENSILE SHEAR TEST RESULTS FOR FM-400 FILM ADHESIVE-
FPL ETCH 7075-T6 BARE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMPERATURE (° F)	PRIMER	
		C-5301	BR-400
		ULTIMATE ⁽³⁾ STRENGTH (PSI)	ULTIMATE ⁽³⁾ STRENGTH (PSI)
FM-400-1 ⁽²⁾	-67	4320	
FM-400-2	-67	4290	
FM-400-3	-67	3880	8160
FM-400-4	-67	4150	7100
FM-400-5	-67	<u>4800</u>	<u>6020</u>
Average		4290 ⁽⁴⁾	7090 ⁽⁵⁾
FM-400-1	Ambient	5260	
FM-400-2	Ambient	4950	
FM-400-3	Ambient	4800	7720
FM-400-4	Ambient	4760	7300
FM-400-5	Ambient	<u>4710</u>	<u>7700</u>
Average		4900 ⁽⁵⁾	7570 ⁽⁵⁾
FM-400-1	180	5210	
FM-400-2	180	5000	
FM-400-3	180	5250	(3)
FM-400-4	180	5160	
FM-400-5	180	<u>4980</u>	
Average		5120 ⁽⁵⁾	
FM-400-1	250	3780	
FM-400-2	250	3280	
FM-400-3	250	2650	5620
FM-400-4	250	3510	5700
FM-400-5	250	<u>3570</u>	<u>5220</u>
Average		3360 ⁽⁵⁾	5510 ⁽⁵⁾
FM-400-1	350	2040	
FM-400-2	350	1880	
FM-400-3	350	1970	4370
FM-400-4	350	1920	4340
FM-400-5	350	<u>1790</u>	<u>4410</u>
Average		1920 ⁽⁵⁾	4370 ⁽⁵⁾

- NOTES: (1) Average bondline thickness was 0.007 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) 180F tensile test on control specimens not required.
(4) Adhesive failures.
(5) Combination adhesive/cohesive failures.

TABLE 26. TENSILE SHEAR TEST RESULTS FOR FM-400 FILM ADHESIVE —
FPL ETCH 7075-T6 BARE AFTER 30 DAY SALT FOG EXPOSURE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMPERATURE (°F)	PRIMER	
		C-5301	BR-400
		ULTIMATE STRENGTH (PSI)	ULTIMATE STRENGTH (PSI)
FM-400-1	Ambient	5300	7310 <u>6400</u> 6860 ⁽³⁾
FM-400-2	Ambient	5340	
FM-400-3	Ambient	5130	
FM-400-4	Ambient	5920	
FM-400-5	Ambient	<u>5560</u>	
Average		5460 ⁽³⁾	
FM-400-1	350	810	2570 <u>3390</u> 2980 ⁽³⁾
FM-400-2	350	930	
FM-400-3	350	1000	
FM-400-4	350	970	
FM-400-5	350	<u>950</u>	
Average		930 ⁽³⁾	

NOTES: (1) Average bondline thickness was 0.007 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Combination adhesive/cohesive failures.

TABLE 27. STRESS RUPTURE TEST RESULTS FOR FM 73 FILM
ADHESIVE 7075-T6 BARE

Specimen Number ⁽¹⁾	Primer	Surface Preparation		Load (Pounds)	Load (% RT Ultimate Strength)	Hours to Failure (4)
		10 Volt Phosphoric Acid Anodize	FPL Etch			
FM-73-1	C-5301 ⁽²⁾	X		1090	40	1714
FM-73-2	C-5301 ⁽²⁾	X		1090	40	1714
FM-73-3	C-5301 ⁽²⁾	X		1090	40	1714
Average						1714
C-FM-73-1	BR-127 ⁽³⁾	X		1060	40	(5)
C-FM-73-2	BR-127 ⁽³⁾	X		1060	40	
C-FM-73-3	BR-127 ⁽³⁾	X		1060	40	
Average						
FM-73-1	C-5301 ⁽²⁾		X	1080	40	466
FM-73-2	C-5301 ⁽²⁾		X	1080	40	261
FM-73-3	C-5301 ⁽²⁾		X	1080	40	529
Average						419
C-FM-73-1	BR-127 ⁽³⁾		X	1085	40	1589
C-FM-73-2	BR-127 ⁽³⁾		X	1085	40	2381
C-FM-73-3	BR-127 ⁽³⁾		X	1085	40	1589
Average						1853

- NOTES: (1) Average bondline thickness was 0.005 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Average BR-127 primer thickness was 0.00018 inch.
(4) Exposure conditions - 120F, 100% RH.
(5) No failure at 2500 hours exposure - ambient temperature tensile strength retention after exposure was 100%.

TABLE 28. STRESS RUPTURE TEST RESULTS FOR FM-400 FILM
ADHESIVE 7075-T6 BARE

Specimen Number ⁽¹⁾	Primer	Surface Preparation		Load (Pounds)	Load % RT Ultimate Strength)	Hours to Failure (4)
		10 Volt Phosphoric Acid Anodize	FPL Etch			
FM-400-1	C-5301 ⁽²⁾	X		835	35	275
FM-400-2	C-5301 ⁽²⁾	X		835	35	275
FM-400-3	C-5301 ⁽²⁾	X		835	35	241
Average						263
C-FM-400-1	BR-400 ⁽³⁾	X		1335	35	152
C-FM-400-2	BR-400 ⁽³⁾	X		1335	35	152
C-FM-400-3	BR-400 ⁽³⁾	X		1335	35	152
Average						152
FM-400-1	C-5301 ⁽²⁾		X	949	35	412
FM-400-2	C-5301 ⁽²⁾		X	949	35	279
FM-400-3	C-5301 ⁽²⁾		X	945	35	279
Average						323
C-FM-400-1	BR-400 ⁽³⁾		X	1325	35	152
C-FM-400-2	BR-400 ⁽³⁾		X	1325	35	152
C-FM-400-3	BR-400 ⁽³⁾		X	1325	35	152
Average						152

- NOTES: (1) Average bondline thickness was 0.007 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Average BR-400 primer thickness was 0.00015 inch.
(4) Exposure conditions — 140°F, 100% RH.

TENSILE SHEAR

-67°F

FM-73
FILM ADHESIVE

BR-127
CONTROL

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

FIGURE 47. FAILED TENSILE SHEAR SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT -67°F-FPL ETCH - PHOSPHORIC ACID ANODIZE

7075-T6 BARE

TENSILE SHEAR

R.T.

FM-73
FILM ADHESIVE

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

1000 HRS
SALT SPRAY

FIGURE 48. FAILED TENSILE SHEAR SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT R.T.-FPL ETCH - PHOSPHORIC ACID ANODIZE

7075-T6 BARE



PHOSPHORIC
ANODIZE



FPL ETCH

1000 HRS
SALT SPRAY

TENSILE SHEAR

180°F

FM-73
FILM ADHESIVE

C-5301
CONTRACT PRIMER

FIGURE 49. FAILED TENSILE SHEAR SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT 180°F-FPL ETCH - PHOSPHORIC ACID ANODIZE

7075-T6 RARE

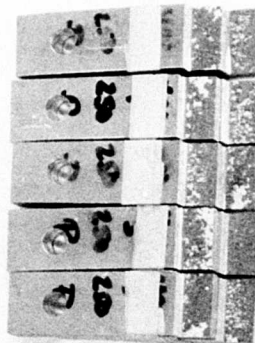
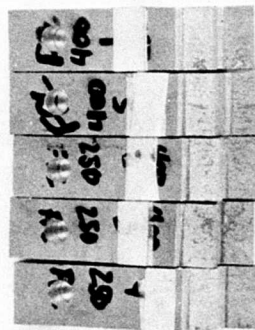
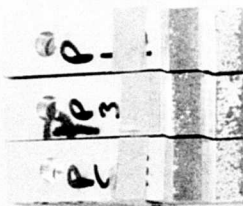
TENSILE SHEAR

250°F

FM-400
FILM ADHESIVE

BR-400
CONTROL

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

7075-T6 BARE

FIGURE 50. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT 250F-FPL ETCH — PHOSPHORIC ACID ANODIZE

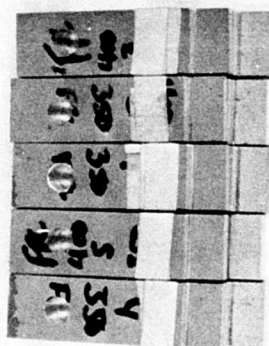
TENSILE SHEAR

350°F

FM-400
FILM ADHESIVE

BR-400
CONTROL

C-5301
CONTRACT PRIMER



PHOSPHORIC
ANODIZE

FPL ETCH

7075-T6 BARE
FIGURE 5L. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT 350°F-FPL ETCH — PHOSPHORIC ACID ANODIZE

TENSILE SHEAR

R.T.

FM-400
FILM ADHESIVE

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

1000 HRS

SALT SPRAY

7075-T6 BARE

FIGURE 52. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT R.T.-FPL ETCH - PHOSPHORIC ACID ANODIZE

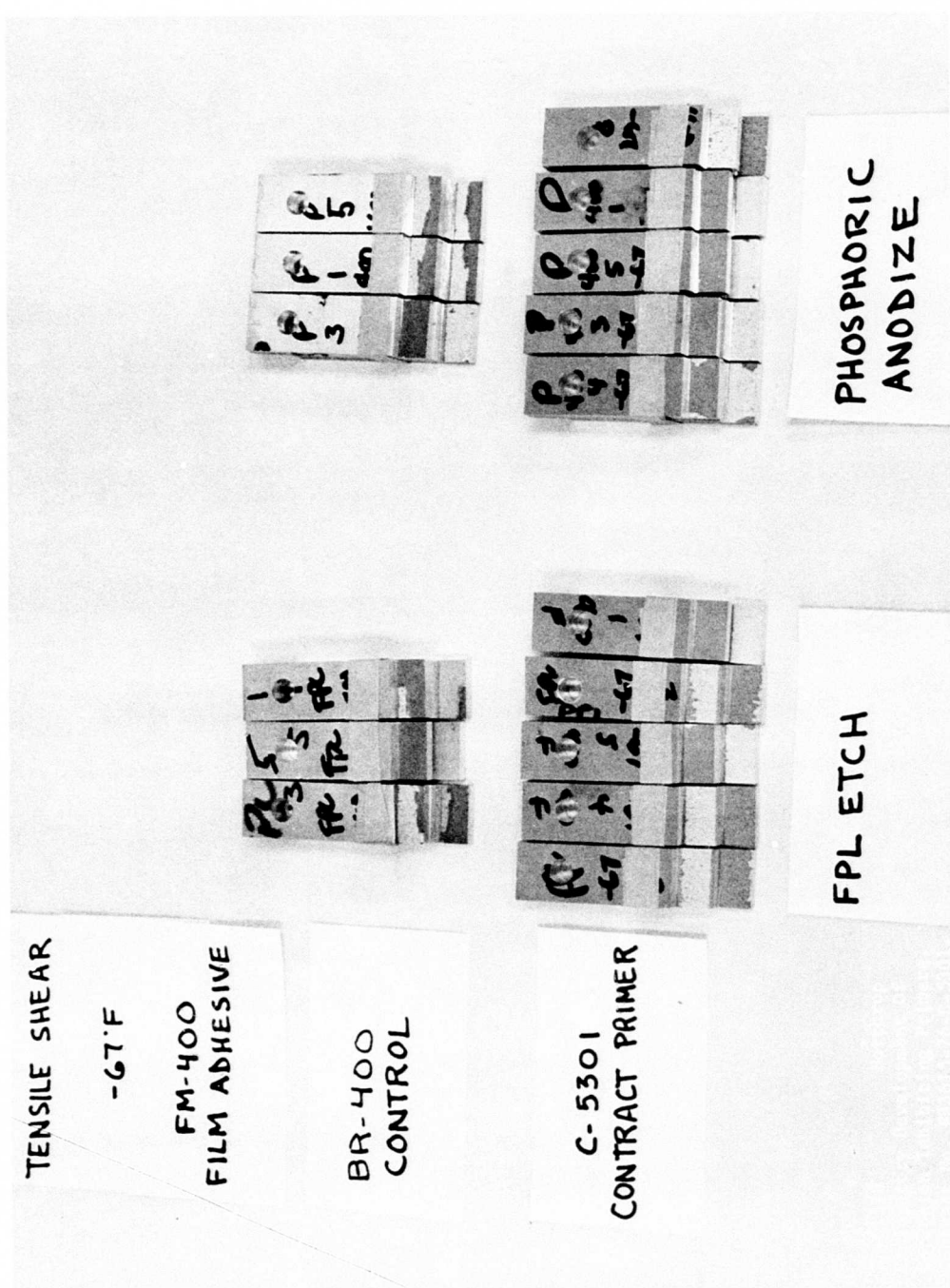


FIGURE 53. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT -67°F-FPL ETCH - PHOSPHORIC ACID ANODIZE

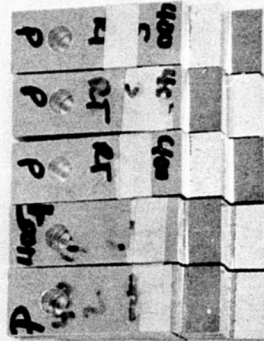
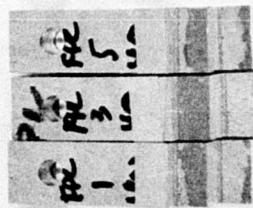
TENSILE SHEAR

R.T.

FM-400
FILM ADHESIVE

BR-400
CONTROL

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

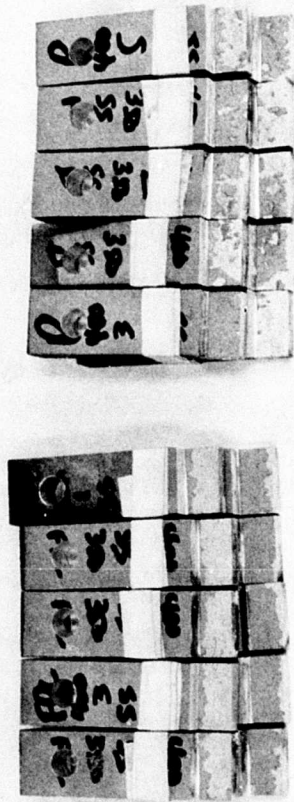
7075-T6 BARE
FIGURE 54. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT R.T.-FPL ETCH - PHOSPHORIC ACID ANODIZE

TENSILE SHEAR

350°F

FM-400
FILM ADHESIVE

C-5301
CONTRACT PRIMER



FPL ETCH

PHOSPHORIC
ANODIZE

1000 HRS
SALT SPRAY

FIGURE 55. FAILED TENSILE SHEAR SPECIMENS FOR FM-400 FILM ADHESIVE
TESTED AT 350°F-FPL ETCH — PHOSPHORIC ACID ANODIZE
7075-T6 BARE

and FM-123-2 adhesive, a much greater (approximately 50%) reduction in tensile shear strengths had been anticipated after the 30 day salt fog exposure. Since 100% cohesive failures were produced by all specimens exposed to the salt fog, it is believed the reduction of the 180F tensile strength is attributable to the degradation of the adhesive as opposed to degradation of the electroprimer or primer/substrate interface. This adhesive degradation is apparent at the elevated test temperature when the adhesive begins to soften. Typical failures are shown in Figures 48 and 49. A control series of test specimens were fabricated, exposed to 30 days salt spray and tensile tested at 180F and ambient temperature. The test data, presented in Table 20 and 22, shows no significant loss in tensile strength of the BR-127/FM-73 adhesive system when exposed to 30 day salt spray compared to the unexposed specimens. Therefore, the 20% reduction in tensile strength of the C-5301/FM-73 adhesive system at 180F after 30 day salt spray exposure may not be attributed exclusively to the softening of the adhesive. The oxide/primer interface of the electroprimed tensile specimens is affected by the salt spray environment but this subtle change in the interface could not be detected by the room temperature tensile test. This change was detected by the more aggressive elevated temperature tensile test.

Tensile shear test results with the FM-400 film adhesive show lower test results at all test temperatures compared to the baseline adhesive primer.

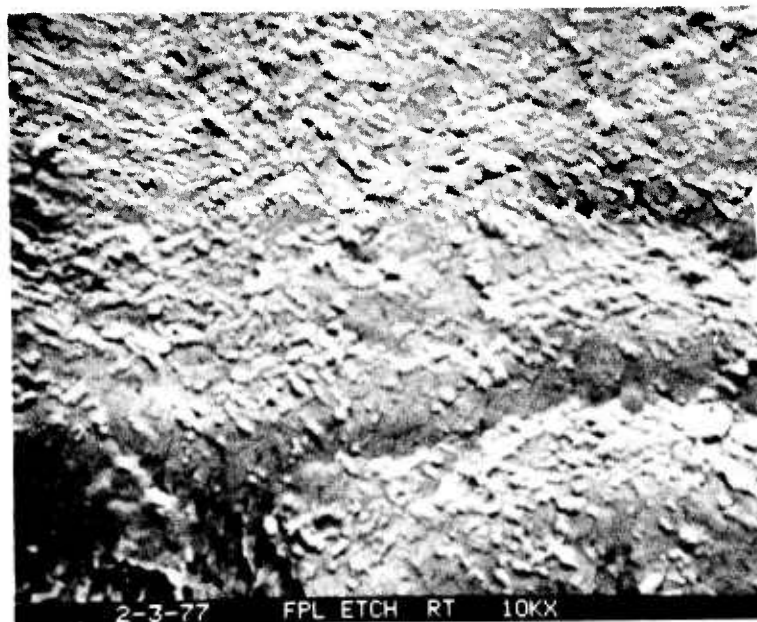
The failure modes for the C-5301 primer showed the failures at the primer/substrate interface whenever adhesive failure occurred. The adhesive failures for the C-5301/FM-400 adhesive system occurred at the -67F and ambient tensile test temperatures. At test temperatures above ambient, the failure modes are primarily cohesive and appear equivalent to the BR-400/FM-400 adhesive system tensile test failures. There was no evidence of any failure at the primer/FM-400 interface. Comparing the tensile strength test results of the C-5301 primer over FPL etch surface treatment to the C-5301 primer over the 10 volt phosphoric acid anodize treatment shows higher strengths are obtained with the 10 volt phosphoric acid anodize surface treatment. This result indicated some effects on the surface treatment oxide are occurring during cathodic electropriming with the application parameters used for this test. Since the normal oxide thickness produced by the FPL etch surface treatment is one-tenth of that produced by the 10 volt phosphoric acid anodize surface treatment, the FPL etch surface treatment reflects the cathodic application effects to a greater extent. Even though the C-5301 provided lower tensile shear strengths compared to

the BR-400 control, the failure modes were comparable in percent cohesive failure in most cases.

It is believed that the contract primer performance can be significantly improved with a more detailed investigation of the application process and formulation development. These considerations are explained in more detail in Section III of this report.

Fracture analyses of the -67F tensile shear specimens indicated in Table 19 and 21 were performed to define the area of failure. Standard 90-degree bend specimens were prepared for SEM analysis from specimens of 7075-T6 bare which had been tensile shear tested. The test samples were taken perpendicular to the tensile shear loading direction from opposite fracture faces of the failed specimens. The samples had been surface treated with FPL etch and 10 volt phosphoric acid anodize prior to electropriming. SEM examination of the -67F failed surfaces showed separation between the electroprimer and the surface of the original oxide layer. In the case of the FPL etch, the layer was approximately 400 Å thick, Figures 56 and 57. The phosphoric acid anodize was approximately 4000 Å thick, Figures 58 and 59. Observation of the surface of the oxide layer did not reveal any fracturing. The primer peeled away from the oxide surface and showed a "replica" of the oxide layer; i.e., the nodular surface, Figure 60. A decided diffusion of the primer into the adhesive was apparent but, physically, there was no primer observed in the oxide layer. The adhesive mode of failure of the -67F tensile test specimens is attributed to the brittle character of the cured C-5301 primer. The mode of failure and the tensile strength of the C-5301/FM-73 adhesive system will be improved by the addition of an elastomer into the electroprimer formulation.

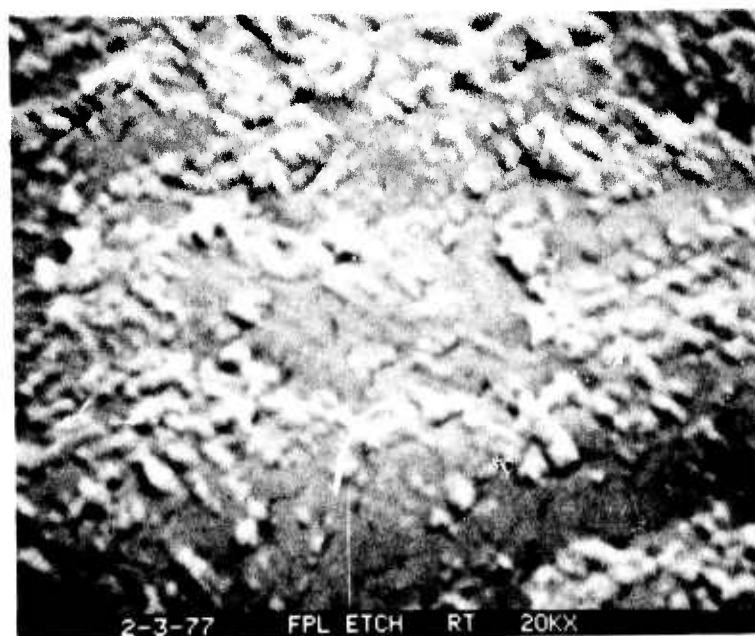
Stress Rupture Tests — The stress rupture tests are defined in Table 18, page 50. The stress rupture test results are presented in Tables 27 and 28. Results of the stress rupture tests utilizing the 250F curing adhesive, FM-73, show the BR-127 control primer producing higher durability test results over both the FPL etch and the 10 volt phosphoric acid anodize surface treatments when compared to the C-5301 primer. The test results utilizing the 350F curing adhesive, FM-400, show the C-530 primer producing higher durability test results over both the FPL etch and the 10 volt phosphoric acid anodize surface treatments when compared to the BR-400 control primer. Care must be taken in the interpretation of these stress rupture test results. A comparative evaluation in durability performance on the C-5301 primer and the control primer, BR-400, cannot be made since the room



7075-T6 BARE

10,000X

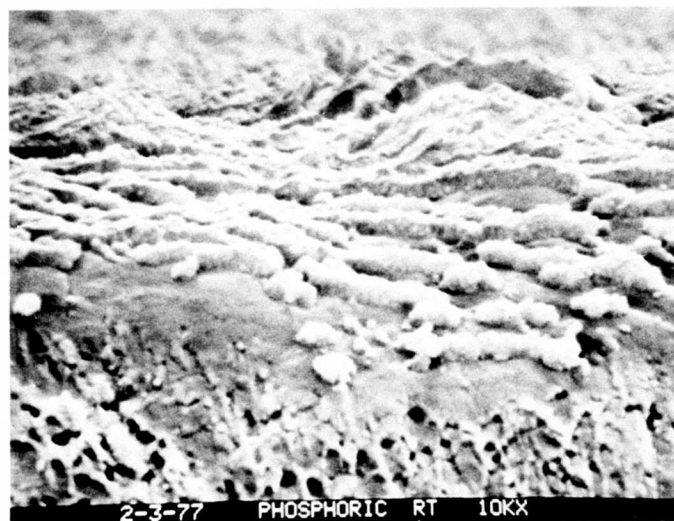
FIGURE 56. SEM PHOTOGRAPH - FPL SURFACE OXIDE IN ADHESIVE FAILURE AREA OF TENSILE SPECIMEN AT 10,000X



7075-T6 BARE

20,000X

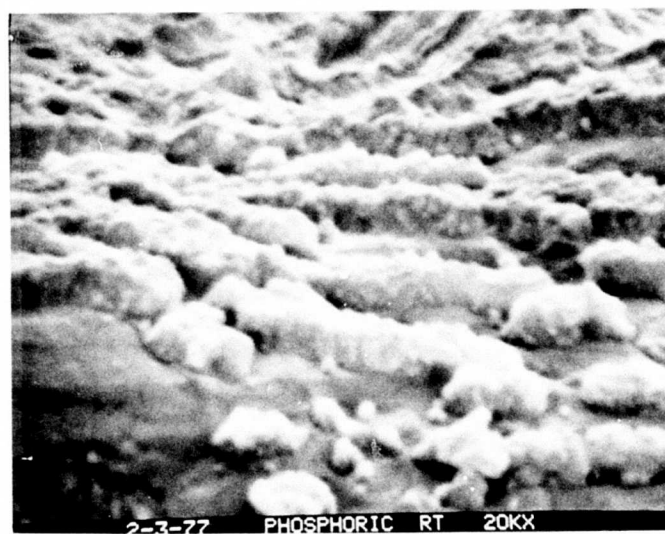
FIGURE 57. SEM PHOTOGRAPH - FPL SURFACE OXIDE IN ADHESIVE FAILURE AREA OF TENSILE SPECIMEN AT 20,000X



7075-T6 BARE

10,000X

FIGURE 58. SEM PHOTOGRAPH - 10 VOLT ANODIZE SURFACE OXIDE IN ADHESIVE FAILURE AREA OF TENSILE SPECIMEN AT 10,000X



7075-T6 BARE

20,000X

FIGURE 59. SEM PHOTOGRAPH - 10 VOLT ANODIZE SURFACE OXIDE IN ADHESIVE FAILURE AREA OF TENSILE SPECIMEN AT 20,000X

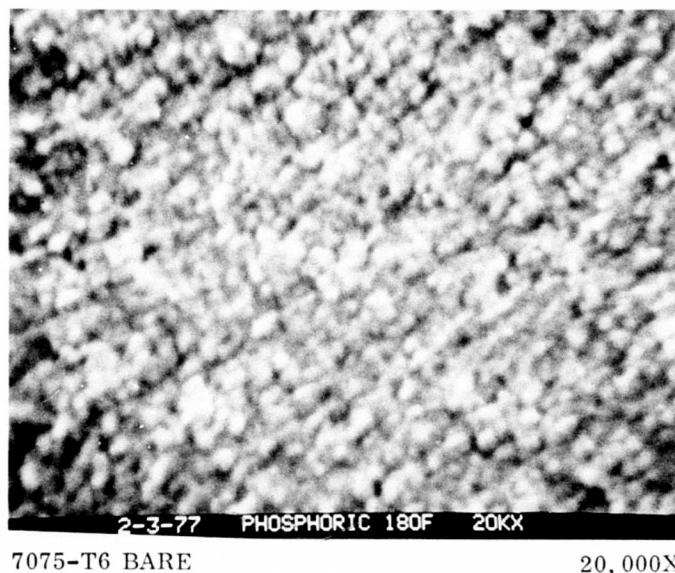


FIGURE 60. SEM PHOTOGRAPH - IMPRESSION OF 10 VOLT ANODIZE OXIDE ON THE ELECTROPRIMER RESIN IN ADHESIVE FAILURE AREA OF TENSILE TEST SPECIMEN.

temperature ultimate strength of the C-5301 primer is substantially lower than the BR-400 control primer.

A comparison of the stress rupture test results of the 250F curing adhesive, FM-73, and the 350F curing adhesive, FM-400, shows the 250F curing adhesive superior in durability test response. The higher performance of the 250F curing adhesive is attributed to its higher plasticity. It is believed that the C-5301 primer is performing in much the same manner and is considered brittle in its presently formulated state. The stress durability response of the C-5301 primer should be significantly improved with further development work by lowering the cure temperature and increasing the plasticity of the primer which will also significantly improve the tensile strength properties of the C-5301/350F curing adhesive system.

T-Peel Tests — The T-Peel tests are defined in Table 18, page 50. The T-Peel test results are presented in Tables 29 and 30.

The T-Peel test results showed the contract primer performed superior to the BR-127 control primer over the 10 volt phosphoric acid anodize at the -67F test temperature.

TABLE 29. METAL/METAL PEEL TEST RESULTS FOR FM-73 FILM
ADHESIVE — 10 V PHOSPHORIC ACID ANODIZE AND FM-123-2 FILM
ADHESIVE — 10 V PHOSPHORIC ACID ANODIZE

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMP (°F)	BARE ALLOY	PRIMER		BARE ALLOY	PRIMER	
			C-5301	BR-127		C-5301	BR-127
			LBS/1" WIDTH	LBS/1" WIDTH		LBS/1" WIDTH	LBS/1" WIDTH
FM-73-1 ⁽²⁾	-67	2024-T3	21	17	7075-T6	11	6
FM-73-2	-67	2024-T3	17	23	7075-T6	13	7
FM-73-3	-67	2024-T3	18	22	7075-T6	22	7
FM-73-4	-67	2024-T3	17	20	7075-T6	23	7
FM-73-5	-67	2024-T3	<u>14</u>	<u>21</u>	7075-T6	<u>16</u>	<u>7</u>
Average			17	20		17	7 ⁽⁴⁾
FM-73-1	Amb	2024-T3	46	47	7075-T6	44	34
FM-73-2	Amb	2024-T3	49	46	7075-T6	44	35
FM-73-3	Amb	2024-T3	47	46	7075-T6	44	34
FM-73-4	Amb	2024-T3	49	47	7075-T6	44	35
FM-73-5	Amb	2024-T3	<u>49</u>	<u>46</u>	7075-T6	<u>44</u>	<u>32</u>
Average			48	46		44	34

FM-123-1	-67	2024-T3	17	23	7075-T6	4	8
FM-123-2	-67	2024-T3	18	24	7075-T6	8	10
FM-123-3	-67	2024-T3	16	24	7075-T6	8	8
FM-123-4	-67	2024-T3	18	25	7075-T6	9	10
FM-123-5	-67	2024-T3	<u>16</u>	<u>23</u>	7075-T6	<u>10</u>	<u>10</u>
Average			17	24		8 ⁽⁴⁾	9 ⁽⁴⁾
FM-123-1	Amb	2024-T3	37	34	7075-T6	26	20
FM-123-2	Amb	2024-T3	38	34	7075-T6	26	20
FM-123-3	Amb	2024-T3	37	34	7075-T6	27	21
FM-123-4	Amb	2024-T3	37	33	7075-T6	26	21
FM-123-5	Amb	2024-T3	<u>36</u>	<u>34</u>	7075-T6	<u>26</u>	<u>20</u>
Average			37	34		26	20

- NOTES: (1) Average bondline thickness was 0.013 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Average BR-127 primer thickness was 0.00018 inch.
(4) Combination adhesive/cohesive failure — all other failures were cohesive.

TABLE 30. METAL/METAL PEEL TEST RESULTS FOR FM-73 FILM ADHESIVE - FPL ETCH AND FM-123-2 FILM ADHESIVE - FPL ETCH

SPECIMEN ⁽¹⁾ NUMBER	TEST TEMP (°F)	BARE ALLOY	PRIMER		BARE ALLOY	PRIMER	
			C-5301	BR-127		C-5301	BR-127
			LBS/1" WIDTH	LBS/1" WIDTH		LBS/1" WIDTH	LBS/1" WIDTH
FM-73-1 ⁽²⁾	-67	2024-T3	16	22	7075-T6	5	7
FM-73-2	-67	2024-T3	17	25	7075-T6	6	7
FM-73-3	-67	2024-T3	21	20	7075-T6	5	8
FM-73-4	-67	2024-T3	20	15	7075-T6	7	8
FM-73-5	-67	2024-T3	<u>19</u>	<u>17</u>	7075-T6	<u>7</u>	<u>8</u>
Average			19	20		6 ⁽⁴⁾	8 ⁽⁴⁾
FM-73-1	Amb	2024-T3	50	47	7075-T6	38	34
FM-73-2	Amb	2024-T3	50	47	7075-T6	37	35
FM-73-3	Amb	2024-T3	50	46	7075-T6	37	32
FM-73-4	Amb	2024-T3	48	48	7075-T6	36	33
FM-73-5	Amb	2024-T3	<u>48</u>	<u>46</u>	7075-T6	<u>36</u>	<u>35</u>
Average			49	47		37	34

FM-123-1	-67	2024-T3	18	23	7075-T6	8	18
FM-123-2	-67	2024-T3	19	22	7075-T6	10	17
FM-123-3	-67	2024-T3	19	22	7075-T6	9	17
FM-123-4	-67	2024-T3	17	23	7075-T6	9	18
FM-123-5	-67	2024-T3	<u>20</u>	<u>23</u>	7075-T6	<u>9</u>	<u>17</u>
Average			19	23		9 ⁽⁴⁾	17
FM-123-1	Amb	2024-T3	37	34	7075-T6	26	21
FM-123-2	Amb	2024-T3	37	34	7075-T6	26	21
FM-123-3	Amb	2024-T3	36	34	7075-T6	26	22
FM-123-4	Amb	2024-T3	36	33	7075-T6	26	20
FM-123-5	Amb	2024-T3	<u>35</u>	<u>32</u>	7075-T6	<u>26</u>	<u>20</u>
Average			36	34		26	21

- NOTES: (1) Average bondline thickness was 0.013 inch.
(2) C-5301 primer thickness was 0.0002 inch.
(3) Average BR-127 primer thickness was 0.00018 inch.
(4) Combination adhesive/cohesive failure - all other failures were cohesive.

In all other test conditions, the C-5301 primer performed equivalent to the BR-127 control primer. The failure modes of the C-5301 and BR-127 on the T-Peel specimens were equivalent in appearance. Typical failure modes of tested T-Peel specimens are shown in Figures 61 through 66.

In summary, based on the test data to date, the overall adhesive bonding assessment has shown the C-5301 primer superior in some tests, and the BR-127 control primer superior in other tests. The performance data for the developed C-5301 electroprimer looks promising, but indicates that further development is required to provide a universal primer superior in all respects to the state-of-the-art hand-sprayed primer.

PHASE IV — COST AND SCALE-UP ASSESSMENT

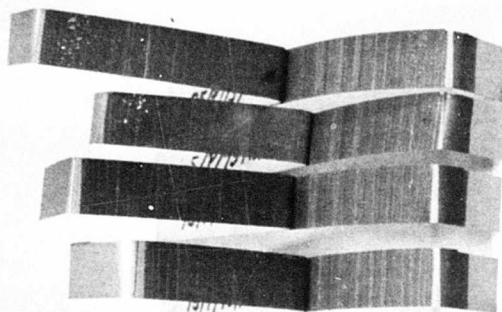
This cost and scale-up assessment had the following objectives:

1. Assess the adaptability of the C-5301 primer to a controlled automated application.
2. Estimate the present application cost of the BR-127 primer.
3. Estimate the costs of an automated application of the C-5301 primer and compare to the BR-127.

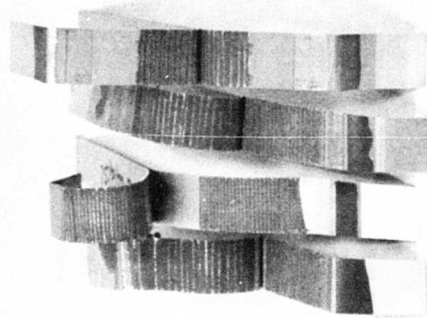
This study uses as a baseline a Northrop engineering assessment of the electroprime process which determined the feasibility and cost effectiveness of installing an automated electroprime line to prime aluminum detail parts for corrosion protection with and without subsequent topcoats. The engineering assessment recommended the procurement and installation of an electropriming facility with a projected savings of 2.0 million dollars based on a 7-year amortization period with a 2.2-year pay-back.

The recommended complete installation, all costs included, was estimated at \$622,000. The assessment was based on the flow of aluminum parts 24-inch wide by 48-inch long or smaller. Larger aluminum parts were not considered in the study. The selection of aluminum parts represents 83% of the Northrop parts using the NAI-1269* primer and approximated 46,000 parts per week. Parts handling methods for cleaning, alodining, and top coating were compared to those proposed for an electroprime facility. Labor content of the present and proposed methods were compared and

*Northrop materials specification - see Appendix B



C-5301
CONTRACT PRIMER



BR-127
CONTROL

MMA-132- PEEL

-67°F

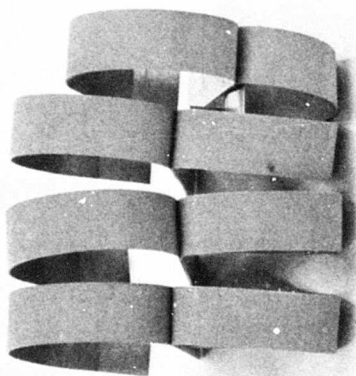
PHOSPHORIC
ANODIZE

7075-T6

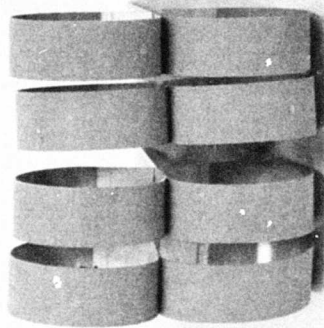
FM-73
FILM ADHESIVE

7075-T6 BARE

FIGURE 61. FAILED T-PEEL SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT -67°F-PHOSPHORIC ACID ANODIZE - 7075-T6 BARE



2024-T3



7075-T6

MMA-132- PEEL

R.T.

FPL ETCH

C-5301

CONTRACT PRIMER

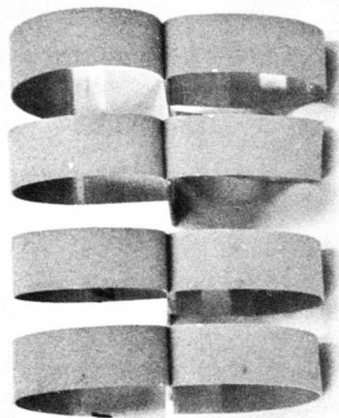
FM-123-2

FILM ADHESIVE

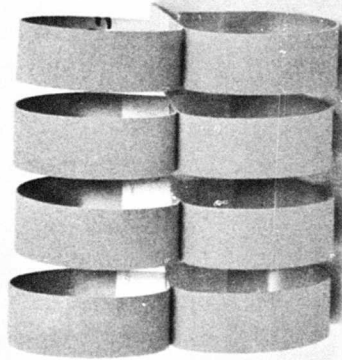
7075-T6 BARE

2024-T3 BARE

FIGURE 62. FAILED T-PEEL SPECIMENS FOR FM-123-2 FILM ADHESIVE
TESTED AT R. T. -FPL ETCH - 7075-T6 BARE AND 2024-T3 BARE



2024-T3



7075-T6

MMA-132- PEEL

R.T.

PHOSPHORIC
ANODIZE

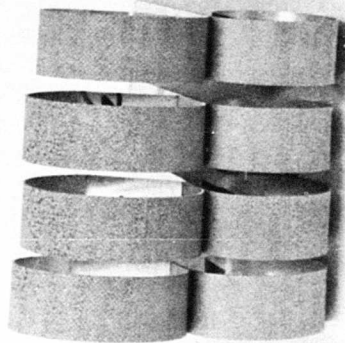
C-5301
CONTRACT PRIMER

FM-123-2
FILM ADHESIVE

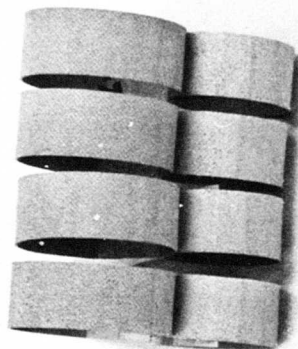
2024-T3 BARE

7075-T6 BARE

FIGURE 63. FAILED T-PEEL SPECIMENS FOR FM-123-2 FILM ADHESIVE
TESTED AT R.T. -PHOSPHORIC ACID ANODIZE - 7075-T6
BARE AND 2024-T3 BARE



BR-127
CONTROL



C-5301
CONTRACT PRIMER

MMA-132- PEEL

R.T.

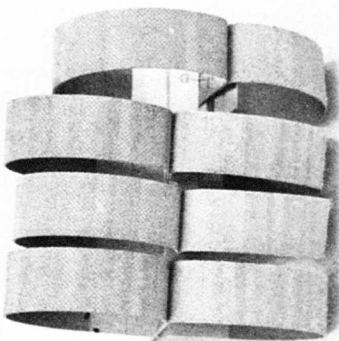
FPL ETCH

7075-T6

FM-73
FILM ADHESIVE

7075-T6 BARE

FIGURE 64. FAILED T-PEEL SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT R.T. - FPL ETCH - 7075-T6 BARE



C-5301
CONTRACT PRIMER



BR-127
CONTROL

MMA-132- PEEL

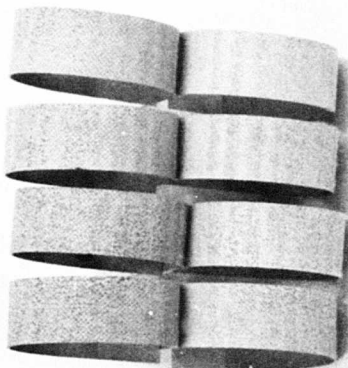
R.T.

FPL ETCH

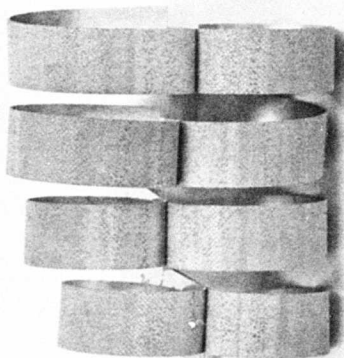
2024-T3

FM-73
FILM ADHESIVE

FIGURE 65. FAILED T-PEEL SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT R.T.-FPL ETCH - 2024-T3 BARE



C-5301
CONTRACT PRIMER



BR-127
CONTROL

MMA-132 - PEEL

R.T.

PHOSPHORIC
ANODIZE

2024-T3

FM-73
FILM ADHESIVE

FIGURE 66. FAILED T-PEEL SPECIMENS FOR FM-73 FILM ADHESIVE
TESTED AT R.T.-PHOSPHORIC ACID ANODIZE - 2024-T3 BARE

a summary of the labor savings given. Material cost reductions made possible by the electroprime process were documented. The major material savings resulted from reduction in the amount of primer used, since electropriming can maintain a controlled film thickness of 0.0002 inch.

Figure 67 shows the proposed process sequence of the automated electropriming line for priming detail aluminum parts for corrosion protection. This line utilizes the Northrop 1330* anodically applied polyester primer for corrosion protection.

Variations of the sequence shown in Figure 67 are in use in the aircraft industry today. These variations in surface preparation include acidified and deionized rinses. The electropriming stage is a total immersion application requiring an approximate 60-second application time with a 2-3 minute rinse and a 30 minute curing cycle.

To adapt the C-5301 electroprimer to automated processing requires the addition of a phosphoric acid anodizing capability shown by the dotted line in Figure 67. Other considerations are the conversion to a cathodic potential which requires a simple electrical rearrangement and verification that all parts in the electropriming tank unit are nonmagnetic. The cathodic bath is subject to iron contamination by dissolution of pump parts, etc., which are not stainless. The Northrop design considered the possibility of cathodic electropriming and designed the proposed electropriming stage exclusively with corrosion resistant alloys (stainless steel).

The engineering study performed by Northrop compared the cost of handspraying a corrosion protection primer which serves as the comparative basis for estimating the application costs of BR-127.

The estimated recurring costs for the BR-127 line assuming 46,000 parts per week are as follows:

Base Labor Costs	\$ 800,000
Base Material Costs	200,000
Base Energy Costs	70,000
Total Base Cost	<u>\$1,070,000/year</u>

*Northrop material specification - see Appendix B.

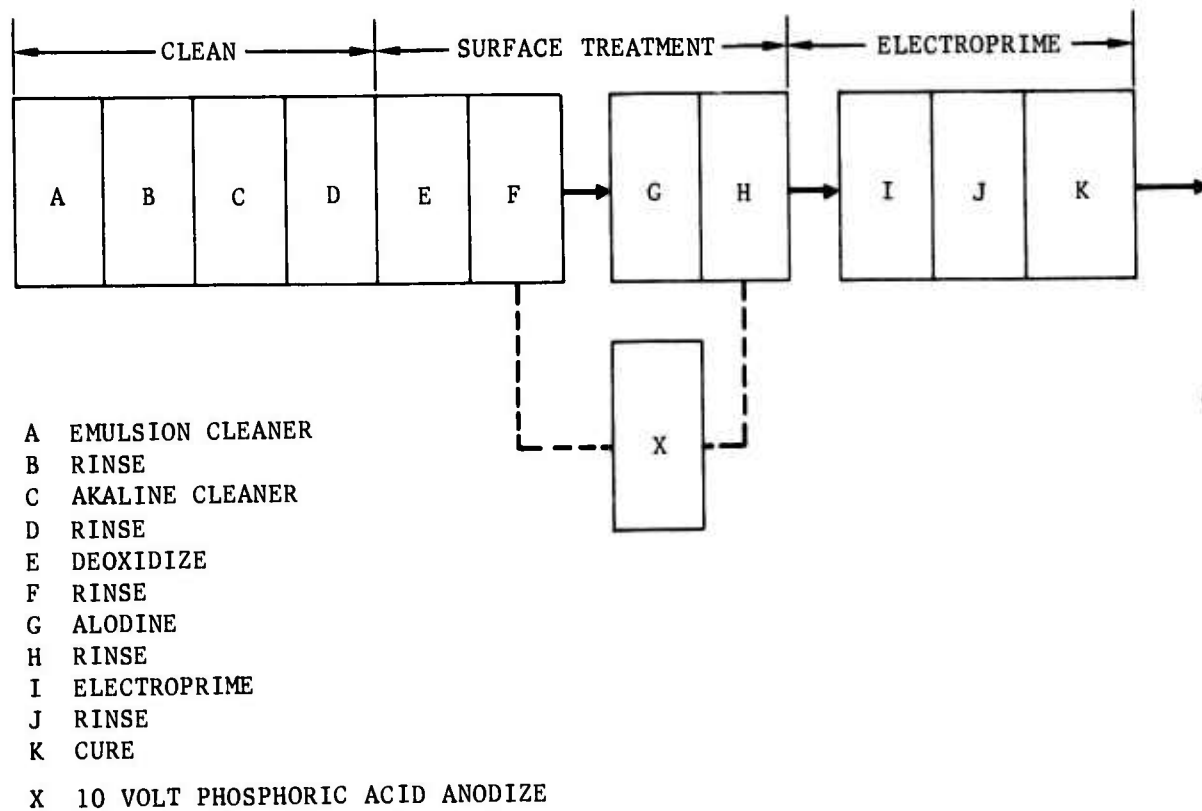


FIGURE 67. PROPOSED NORTHROP NAI 1330
ELECTROPRIME PROCESS LINE

Again using the basic electropriming line with the addition of the phosphoric acid anodize, the estimated recurring costs for a C-5301 electropriming line assuming 46,000 parts per week would be as follows:

Base Labor Costs	\$ 535,000
Base Material Costs	80,000
Base Energy Costs	30,000
Total Base Cost	<u>\$ 645,000/year</u>

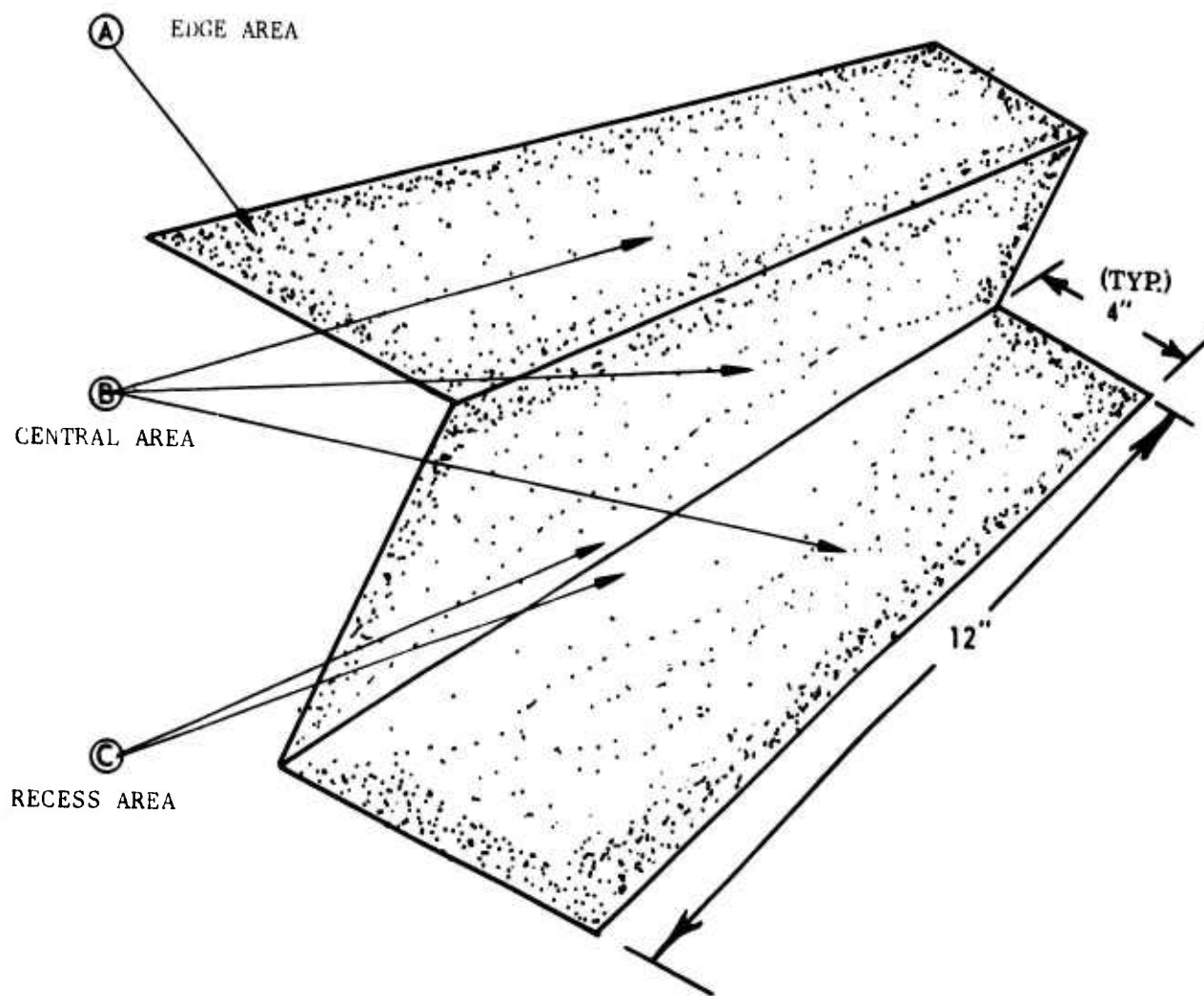
The recurring cost differential, or potential yearly savings amounts to \$425,000 based on a load of approximately 46,000 parts per week. Based on estimated used in the referenced study, approximately \$800,000 would be required to install a C-5301 electroprimer system capable of treating up to 46,000 parts per week. The maximum part size would be 72 inches in length and 48 inches in width.

In summary, a projected annual savings of \$425,000 can be realized utilizing the C-5301 electroprime compared to the conventional hand sprayed BR-127. An investment of approximately \$800,000 would be required to implement a completely automated C-5301 cathodic electropriming facility.

PHASE V — UNIFORMITY AND REPRODUCIBILITY ASSESSMENT

To comparatively evaluate the hand-spray application method of priming versus the electropriming application method, relative to uniformity and reproducibility, a fairly simple part configuration was designed. A sketch of the part is presented in Figure 68. Eight specimens of this configuration were fabricated. Four of the specimens were hand sprayed with BR-127 adhesive primer in a production priming facility on four different days, and four were electroprimed in the laboratory's 20 gallon electropriming cell. The laboratory electropriming cell is a replica of production type automated units. Results of this comparative study are presented in Table 31.

The test data shows all specimens met the average adhesive primer film thickness requirement of 0.1 - 0.3 mil. However, out of 120 thickness determinations taken on the hand sprayed specimens, 28 individual readings were out of the required 0.1 - 0.3 mil range; 8 determinations below and 20 determinations above the designated range. All film thickness determinations on the electroprimed specimens fell well within the required range. The areas on hand sprayed substrates which are most likely to be out of the tolerance range are the edges, whether the part be of flat stock



ELECTROPRIME METHOD

HAND SPRAY METHOD

- (A) Uniform Primer Applied
- (B) Uniform Primer Applied
- (C) Uniform Primer Applied

- Excess Primer Applied
- Average Primer Applied
- Minimal Primer Applied

FIGURE 68. UNIFORMITY AND REPRODUCIBILITY SPECIMEN CONFIGURATION

TABLE 31. UNIFORMITY AND REPRODUCIBILITY TEST RESULTS FOR
HAND SPRAYED VERSUS ELECTROPRIMED SUBSTRATES

SPECIMEN	PRIMER	PRIMER THICKNESS (MIL)			
		HIGH	LOW	RANGE	AVERAGE
1	BR-127	0.38	0.04	0.34	0.20
2	BR-127	0.38	0.06	0.32	0.23
3	BR-127	0.38	0.11	0.27	0.24
4	BR-127	0.36	0.10	0.26	0.27
1	C-5301	0.20	0.16	0.04	0.18
2	C-5301	0.20	0.15	0.05	0.18
3	C-5301	0.19	0.15	0.04	0.17
4	C-5301	0.19	0.14	0.05	0.17

or compound curvature. This is due to the airflow pattern of the material around the parts being coated. The film thickness is generally high in those areas close to the edges. The electroprimer is far superior relative to reproducibility even though both methods are acceptable. The controlling factor in the electropriming process for film thickness is the applied potential. For this assessment, a 30 volt potential was used. The film will be reproduced by any operator using the 30 volt application potential.

The process can be further controlled by "locking in" the 30 volt application potential on the control panel. In hand sprayed primers, day-to-day reproducibility is operator-dependent and is a developed art.

SECTION III
CONCLUSIONS AND RECOMMENDATIONS
FOR FUTURE WORK

CONCLUSIONS

The objective of this program was to develop corrosion inhibiting primers for adhesive bonding of aluminum alloys which are more reproducible and less costly to apply than current state-of-the-art corrosion inhibiting primers. The primers shall be compatible as a minimum goal with 250F curing 180F service capability state-of-the-art structural adhesives, and desirably with 350F service adhesives, and also aircraft paint primers and paints (primarily exterior). Further, the primers shall have a low total or "in-place" cost, based on materials processes and quality assurance considerations. They shall be easy to process, uniform in coverage, reproducible, adaptable to compound curvature of parts and also to automated processing.

The polyester, acrylic, and epoxy electropriming resin systems were investigated. The three resin systems all produced acceptable wedge test results over the 10 volt phosphoric acid anodize surface treatment comparable to the BR-127 baseline primer; i.e., crack extension growth of less than 0.25 inch in a 24 hour exposure.

The polyester electropriming resin system was unable to produce an acceptable wedge test result over an FPL etch surface treatment, even though it was demonstrated that significant improvement in performance could be achieved with an application potential less than 15 volts. The acrylic electropriming resin system produced acceptable results over both the 10 volt phosphoric acid anodize, and the FPL etch surface treatments. However, incompatibilities between the acrylic electropriming resin and the modified epoxy film adhesive, FM-123, were noted at ambient and 180F tensile shear tests. The tensile shear test results were low, results scattered, and the failure modes indicated as high as 90% adhesive failure occurring between the primer and adhesive.

Acceptable wedge test, tensile shear test, and paint compatibility performance was achieved by the modified epoxy electroprimer during screening test evaluations. The anodic electropriming process was investigated. Instrumental analysis showed

increasing anodic application potentials increased the primer/substrate interface oxide thickness and character, and the increase in oxide thickness detrimentally affected wedge test performance. Instrumental analysis of the cathodic electrodeposition process verified that the oxide interface did not change form during cathodic electropriming over FPL etch and 10 volt phosphoric acid anodize control surface preparations. Further, SEM analyses showed the surface treatment oxide did not change in thickness or appearance when cathodic electropriming with the C-5301 contract primer. Based on the above findings, it was concluded that the cathodically deposited modified epoxy electroprimer, C-5301, was the best resin/process available to meet the program objectives. Efforts were made to lower the cure schedule of the initial modified epoxy formulation. The initial modified epoxy electroprimer cured at 400F for 30 minutes. Defining a more acceptable class of crosslinker (catalyst) lowered the cure requirement of the epoxy to 375F and, subsequently, to 345F for 30 minutes. Acceptable wedge test results were also obtained using a cure schedule of 325F for 60 minutes. Based on these developments, it is concluded that the cathodically applied modified epoxy electroprimer cure schedule can be reduced below 300F. It is projected that a 250F/275F cure schedule can be achieved, with further primer resin formulation efforts.

The developed contract electroprimer C-5301 performed acceptably in evaluation tests relative to paint systems compatibility and provided mixed results relative to bondability. The T-peel test results with the C-5301 contract primer were higher than the baseline BR-127 primer. With the FM-73 adhesive, the ambient and elevated temperature tensile shear test results with the C-5301 were higher than the BR-127 control, but comparatively lower with the FM-400 film adhesive. Based on these test results, it is concluded that the C-5301 electroprimer has not been completely optimized relative to the aspects of bonding with 350F curing adhesives.

The electropriming application method produced films reproducible within ± 0.00001 inch in thickness with a film thickness uniformity of ± 0.00003 inch. The hand sprayed BR-127 produced films reproducible within ± 0.00004 -inch average film thickness with a film uniformity of ± 0.00014 inch. It is concluded that electropriming is far superior to hand sprayed primers relative to uniformity and reproducibility and is independent of part configuration. Further, the film thickness uniformity of hand-sprayed primer is grossly affected by part configuration, whereas electropriming assures film thickness uniformity on the most complex parts.

The intrinsic advantages of the electropriming application method over the current state of-the-art hand sprayed priming method are overwhelming. Comparatively these advantages are:

1. The electropriming method is cost effective relative to labor and material. The electropriming method is readily adaptable to automated processing thereby significantly reducing the labor required for parts handling. Electropriming is material usage effective, with efficiencies over 95 percent! The hand spray priming method is labor intensive and is inefficient in material usage, with a 50% overspray loss and a subsequent solvent evaporation loss from the applied film. The net material usage efficiency of hand sprayed coatings is only 25 percent.
2. Electropriming provides a continuous processing sequence from precleaning through primer cure. The net effect eliminates shop contamination between surface preparation and priming and thereby eliminates oxide changes in the surface preparation which result from atmospheric exposure. The hand spray priming method subjects parts to latent organic shop contaminants in the time dwell between surface preparation and priming and also provides the atmosphere for random oxide changes in the surface preparation. The susceptibility to changes in the surface preparation oxide must be emphasized as a major concern since it explains the cause of various random bonding failures which heretofore have not been explainable. Even the latent sulfur from heating fuels used in some manufacturing plants can and do sulfonate the aluminum oxide surface preparation and provides a failure mechanism for subsequent adhesive bonds. Electropriming effectively eliminates these problem areas with a continuous process sequence.
3. The electropriming method provides total coverage of complex shapes. This advantage becomes increasingly significant when considerations are given to the complexity of detail parts on the interior of the aircraft. All areas of the complex parts are protected including recesses, holes, and "blind" areas. Evaluating two priming methods in a laboratory test on flat panels fails to correlate to actual production items and may indicate equivalency in the two priming methods. Hand sprayed detail aircraft parts will have voids and "lean" areas due to limitations of the spraying method. Evaluation of the two methods on actual complex production parts will conclusively show the superiority of the electropriming method which provides total coverage of all parts.

4. The electropriming application method provides reproducible coatings. In a manufacturing process line, it will reproduce the same film independent of time and independent of an operator's developed art. Hand sprayed primers are totally operator dependent. The developed technique of the operator and his attitude to utilize the proper techniques, all contribute to the quality, or lack thereof, of the finished parts.
5. The electropriming application method provides a uniform primer film. The thickness of the deposited film is controlled by the pre-set application potential which provides a specific thickness. The hand sprayed primer thickness will not only vary from one spray booth to another due to air flow volume and pattern, but will also vary due to the "visibility gage" of one operator to another. The other aspect of controlled uniformity is relative to process inspection wherein the operator primes parts to satisfy the visual interpretation of the process inspector rather than the controlling process document.
6. The electropriming method is ecologically preferred. All of the primer material put in the process tank is deposited on the work piece as opposed to hand sprayed primers which spew 75% of all material emanating from the paint gun up the flue and into the atmosphere.
7. The electropriming method is desirable from a safety aspect. The electroprimer is a water reducible coating, and as such does not require solvents for dilution and clean-up. The current aircraft primers are solvent reducible coatings, and intrinsically are a safety and health hazard. All aircraft plants are plagued by the voluminous demand for solvents required for painting and priming.
8. The electropriming application method provides corrosion protection of detail aluminum parts at a uniform film thickness of 0.2 mil. Hand sprayed primers are applied to 0.5 to 1.2 mil in thickness. Considering the total surface area of all parts in the aircraft to be primed, the electroprimer film provides a significant weight saving.

Considering the overwhelming advantages of the electropriming process and the promising results of this contract effort, it is the author's opinion that the entire aircraft industry should exploit this process in an expeditious manner to provide the civilian and military aircraft market with higher performance, most effective aircraft.

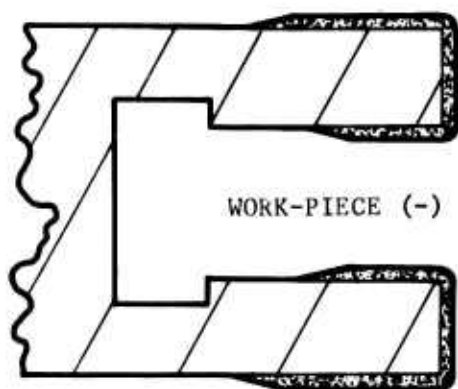
RECOMMENDATIONS FOR FUTURE WORK

Based on the encouraging results of this test program, the following recommendations for future work are made:

1. Develop a 250F curing cathodically applied modified epoxy electroprimer.
2. Optimize the modified epoxy electroprimer for adhesive bonding by defining the best class and concentration of the key components in the electropriming formulation.
3. Characterize the electroprimer/substrate interfacial oxide after electropriming.
4. Develop a new surface treatment for aluminum substrates which is responsive to automated processing (the 10 volt phosphoric acid anodize surface requires a 20 minute immersion time).
5. Develop methods for electropriming honeycomb core to provide a complete corrosion resistant adhesive bonded aluminum structure.

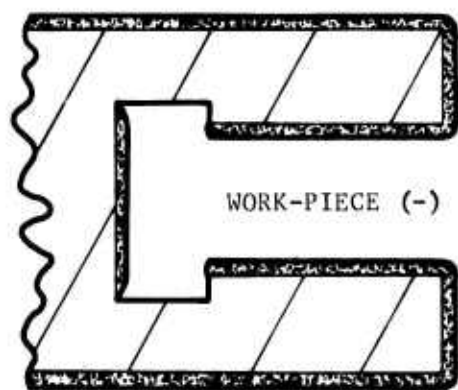
APPENDIX A

MECHANISM OF FILM FORMATION
AND SCHEMATIC DIAGRAM OF CATHODIC ELECTROPRIMING



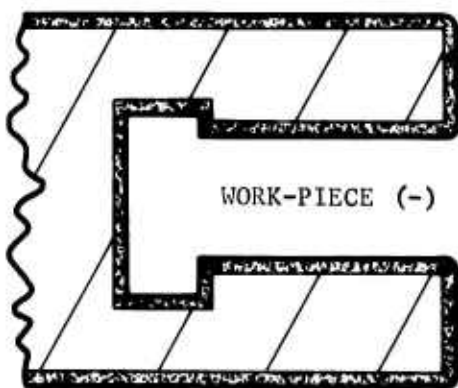
STAGE 1

DEPOSITION STARTS ON EDGES AND SURFACES CLOSEST TO ANODE



STAGE 2

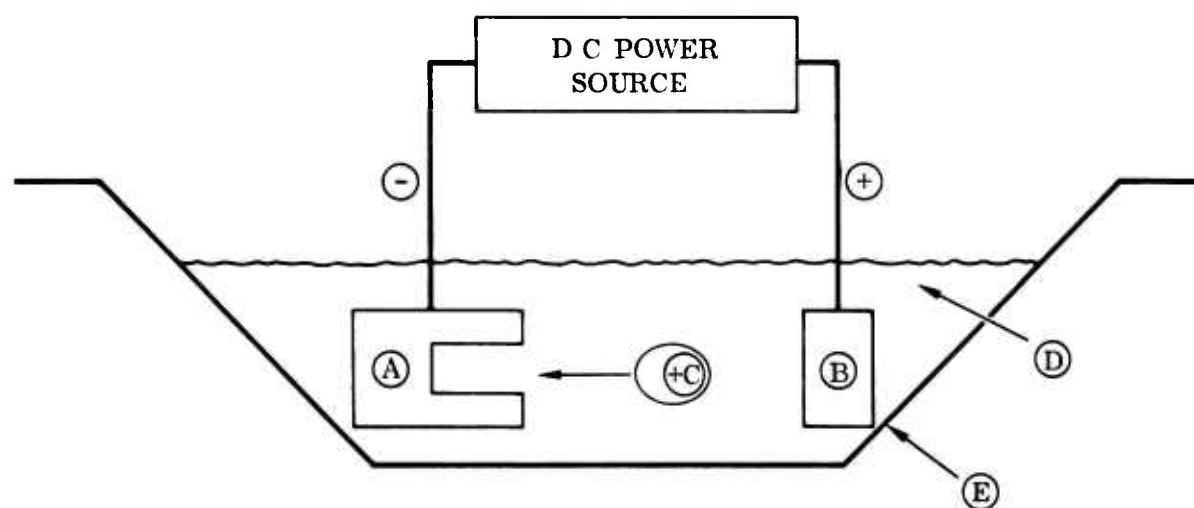
INSULATIVE NATURE OF DEPOSITED FILM RESULTS IN DEPOSITION ON RECESSED AREAS



STAGE 3

FINAL STAGE RESULTS IN COMPLETE COVERAGE AND DENSIFICATION OF FILM

MECHANISM OF FILM FORMATION ON IRREGULAR SURFACES



- (A) - WORKPIECE - CATHODE (-)
- (B) - AUXILIARY - ANODE (+)
- (C) - ELECTROPRIME RESIN
- (D) - WATER SOLUTION OF RESIN SYSTEM
- (E) - ELECTROPRIMING TANK

SCHEMATIC DIAGRAM OF
CATHODIC ELECTROPRIMING

APPENDIX B

NORTHROP CORPORATION

MATERIAL SPECIFICATION

AND PAINT SYSTEMS COMPATABILITY TEST METHODS

RESPONSIBLE ENGINEER <i>R. H. H. H.</i>	NORTHROP Northrop Corporation Aircraft Division MATERIAL SPECIFICATION	NAI-1269	DATE 2 May 1972 RELEASE EO D56942 CODE IDENT. NO. 76823	C REVISION
AIRCRAFT DIVISION <i>E. E. E.</i>				
SPECIFICATION CONTROL <i>B. A. A.</i>				
PROJECT OFFICE <i>T. H. H.</i>				

TITLE: FLUID RESISTANT CATALYZED PRIMER

(This revision supersedes Material Specification NAI-1269, Revision B, dated 10-19-71. Technical changes from the previous issue are marked TC in the outer margin.)

1. SCOPE

- 1.1 This specification establishes the requirements for an air dry epoxy primer as a protective, corrosion inhibiting, fluid resistant undercoat to be used alone or in conjunction with a topcoat.
- 1.2 The primer, as controlled by this specification, is a 2 part material, air dry type, for interior or exterior use on aerospace vehicles. It is to be used as a corrosion inhibiting coating or as a corrosion inhibiting undercoat.
- 1.3 Primers meeting the requirements of this specification meet the requirements of the Los Angeles County Air Pollution Control District, Rule 66, for materials to be used within Los Angeles County, California.

2. APPLICABLE DOCUMENTS

- 2.1 The following publications of the issue in effect on the date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

2.2 Government

2.2.1 Specifications

- | | |
|---------------------|---|
| 2.2.1.1 CCC-C-419 | Cloth, Duck Cotton, Unbleached, Plied Yarns Army and Numbered |
| 2.2.1.2 QQ-A-250/4 | Aluminum Alloy 2024, Plate and Sheet |
| 2.2.1.3 QQ-A-250/5 | Aluminum Alloy Alclad 2024, Plate and Sheet |
| 2.2.1.4 QQ-A-250/12 | Aluminum Alloy 7075, Plate and Sheet |
| 2.2.1.5 QQ-A-250/14 | Aluminum Alloy 7178, Plate and Sheet |
| 2.2.1.6 QQ-M-44 | Magnesium Alloy Plate and Sheet (AZ31B) |
| 2.2.1.7 TT-E-781 | Ethylene Glycol Monoethyl Ether |
| 2.2.1.8 TT-I-735 | Isopropyl Alcohol |
| 2.2.1.9 TT-M-261 | Methyl Ethyl Ketone, Technical |
| 2.2.1.10 TT-N-95 | Naptha, Aliphatic |
| 2.2.1.11 TT-S-735 | Standard Test Fluids; Hydrocarbon |
| 2.2.1.12 TT-T-266 | Thinner, Lacquer |
| 2.2.1.13 MIL-C-5541 | Chemical Conversion Coatings on Aluminum and Aluminum Alloys |
| 2.2.1.14 MIL-H-5606 | Hydraulic Fluid, Petroleum Base; Aircraft Missile, and Ordnance |
| 2.2.1.15 MIL-L-7808 | Lubricating Oil, Aircraft Turbine Engine, Synthetic Base |

TC

NORTHROPNorthrop Corporation
Aircraft DivisionMATERIAL SPECIFICATION: NAI-1269
Revision C

DATE 2 May 1972

- 2.2.1.16 MIL-A-8625 Anodic Coatings, for Aluminum and Aluminum Alloys
- 2.2.1.17 MIL-S-8802 Sealing Compound, Temperature-Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High Adhesion
- 2.2.1.18 MIL-C-9084 Cloth, Glass, Finished, for Polyester Resin Laminates
- 2.2.1.19 MIL-R-9300 Resin, Epoxy, Low-Pressure Laminating
- 2.2.1.20 MIL-M-45202 Magnesium Alloys, Anodic Treatment of
- 2.2.1.21 MIL-S-83315 Sealing Compound, Aluminum Structure, Pressure and Weather Sealing, Low Density
- 2.2.2 Standards
 - 2.2.2.1 Federal Test Method Standard No. 141 Paints, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling and Testing
 - 2.2.2.2 Federal Standard No. 595 Colors
- 2.2.3 Industry
 - 2.2.3.1 ASTM D268 Sampling and Testing Lacquer Solvents and Diluents
 - 2.2.3.2 ASTM D522 Elongation of Attached Lacquer Coatings With the Conical Mandrel Test Apparatus

3. REQUIREMENTS**3.1 Qualification**

- 3.1.1 The solvent structure of the primer supplied to this specification shall meet all requirements of the Los Angeles County Air Pollution Control District, Rule 66, for materials to be used within Los Angeles County, California.
- 3.1.2 Qualification Prior to Production
 - 3.1.2.1 This specification requires products qualification prior to acceptance of production orders. All materials furnished by a supplier to this specification shall meet all requirements listed herein, regardless of the extent of quality control tests.
 - 3.1.2.2 Product qualification is granted only by Aircraft Division Materials Engineering.
 - 3.1.2.3 No change in raw materials or method of manufacture shall be made subsequent to qualification without notification and prior approval in writing from Aircraft Division Materials Engineering. Requalification of a revised material or method of manufacture shall be required.
 - 3.1.2.4 After qualification, all subsequent shipments shall be from the same manufacturing plant that submitted the approval qualification sample. Any other plant, although from the same company, shall be approved before material is shipped from that plant.
- 3.1.3 Temporary Qualification
 - 3.1.3.1 Upon successful completion of the qualification tests specified herein, except stability (3.3.5), a material may be given temporary approval and be listed on the Qualified Products List to this specification.
 - 3.1.3.2 Final approval shall be based on successful completion of the test for stability.

3.2 Materials

- 3.2.1 Vehicle - The vehicle used in the formulation of this primer shall be an amine cured epoxy as determined in accordance with 4.7.20.

NORTHROP

Northrop Corporation
Aircraft Division

MATERIAL SPECIFICATION: NAL-1269
Revision C

DATE 7 May 1972

- 3.2.2 Application - The primer base material plus catalyst and thinner (if necessary) are each to be mixed in the volume ratio specified by the manufacturer to produce a suitable viscosity for application by spray, brush, or flowcoat equipment.
- 3.2.3 Appearance - The material shall be free of skins, lumps, grit, and all foreign contaminants and shall be easily mixed to a smooth homogeneous condition as received, after mixing, during pot life, and after one year of storage. The material shall be free of skins, lumps, grit, and all foreign contaminants after application.
- 3.2.4 Odor - The odor of the primer (base or catalyst) shall not cause discomfort to operator applying the materials.
- 3.2.5 Toxicity - Materials meeting the requirements of this specification shall have no adverse effect on the health of personnel during use or curing when used for the intended purpose.

3.3 Physical Properties

3.3.1 Weight Per Gallon

- 3.3.1.1 The weight per gallon of each of the components shall not vary by more than ± 0.2 pound per gallon when tested in accordance with 4.7.1. The thinner (if required) shall not vary by more than ± 0.1 pound per gallon. The established value for the base material shall be 9.0 to 10.5 pounds per gallon, and the catalyst shall be 6.7 to 7.4 pounds per gallon.
- 3.3.1.2 The actual values established during qualification of each material shall be listed on the Qualified Products List.

3.3.2 Nonvolatile

- 3.3.2.1 The nonvolatile content of the base material and catalyst shall not vary from the established percentage by more than ± 2 percent. The nonvolatile content of each of the components shall be determined separately in accordance with 4.7.2. The nonvolatile content of the thinner (if required) shall not exceed 5 milligrams per 100 milliliters when tested in accordance with 4.7.2.
- 3.3.2.2 The actual values established during qualification of each material shall be listed on the Qualified Products List.

- 3.3.3 Viscosity - The viscosity of the base component mixed with catalyst and thinner (when specified by manufacturer) shall be 16 seconds ± 0.5 when tested in accordance with 4.7.3.

- 3.3.4 Pot Life - There shall be no evidence of skinning, gelling, or seeding after the mixed material has been allowed to stand at room temperature for 15 to 17 hours in accordance with 4.7.4. If necessary, the material may be restored to the established viscosity by the addition of not more than 20 percent (by volume) thinner. The thinned material shall meet the spraying requirement of the established viscosity. The resulting coating shall have a satisfactory surface appearance and shall meet the following requirements.

- a. Dry time (3.3.7)
- b. Surface appearance (3.2.3)
- c. Color (3.3.8)
- d. Metal anchorage (3.4.5)
- e. Distilled water immersion (3.4.4)
- f. Humidity (3.4.1)

- 3.3.5 Stability - Material that has been stored in a full container at 60 to 90 F for one year from date of manufacture shall meet the requirements specified in 4.4. Testing shall be conducted after 12 months storage.

- 3.3.6 Settling - The solids content of the mixed material shall be completely redispersed and suitable for application upon shaking after having been permitted to stand undisturbed for 16 to 17 hours when tested in accordance with 4.7.5.

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Aircraft DivisionMATERIAL SPECIFICATION: NAI-1269
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3.3.7 Dry Time - The applied coating shall air dry within the following maximum time periods when tested in accordance with 4.7.6.

- a. Dust free 15 minutes
- b. Tack free 2 hours
- c. Hard 4 hours

3.3.8 Color - The color of the primer shall be yellow-green in the range from yellow approximating Federal Standard No. 595, Color 23685, to green approximating Color 34138, characteristic of the chromate pigments used.

3.4 Film Properties

3.4.1 Humidity Resistance - The primer shall show no loss of adhesion, blistering, or softening of the film after 720 to 750 hours in condensing humidity at $120\text{ F} \pm 2$ when tested in accordance with 4.7.8. Blistering that extends less than 0.125 inch from the panel edge shall not be considered failure.

3.4.2 Impact Resistance - The primer shall not crack or lose adhesion when subjected to an impact of 50 inch pounds on the coated surface and 30 inch pounds on the reverse side of the panel when tested in accordance with 4.7.9.

3.4.3 Corrosion Resistance - The applied primer shall protect aluminum substrate from corrosion for a minimum of 1000 hours and magnesium substrate for a minimum of 500 hours when exposed to 5 percent salt spray and tested in accordance with 4.7.10. Corrosion within 0.125 inch of the scribe mark and panel edge shall not be considered failure.

3.4.4 Fluid Resistance - The applied primer shall withstand immersion in the following fluids at $75\text{ F} \pm 5$ for the time specified without blistering, loss of adhesion, softening, or other film failures when tested in accordance with 4.7.11. Blistering which is obviously caused by contamination, such as fingerprints or marring of the panel due to scratches, and those blisters occurring within 0.25 inch of a panel edge or hole shall not be cause for rejection.

- a. Distilled water 7 days
- b. MIL-H-5606 Fluid 7 days
- c. MIL-L-7808 Jet engine oil 7 days

3.4.5 Metal Anchorage - The applied primer shall show satisfactory adhesion to chemically treated metal with no flaking and no cracking beyond 1/2 inch from the 1/8 inch end of a conical mandrel when tested in accordance with 4.7.12.

3.4.6 Heat Resistance - The applied primer shall show no blistering, loss of adhesion, or other evidence of film failure when tested in accordance with 4.7.13. Discoloration and cracking without adhesion loss shall not be cause for rejection.

3.4.7 Low Temperature Resistance - The applied primer shall show no loss of adhesion or other evidence of film failure when tested in accordance with 4.7.14.

3.4.8 Sealant Compatibility

3.4.8.1 The applied primer shall be compatible with MIL-S-8802 and MIL-S-83315 sealing compound.

3.4.8.2 When tested for compatibility with sealants, the peel strength of the sealant shall average at least 20 pounds per inch peel with no value lower than 15 pounds for any one specimen. The separation shall be a minimum of 95 percent in cohesion of the sealant, disregarding areas of sealant to cloth failure, when tested in accordance with 4.7.15.

3.4.9 Finish System Compatibility - The applied primer from any qualified supplier shall be compatible with the primer of any other qualified supplier when applied as part of a finish system and tested in accordance with 4.7.16.

3.4.10 Repairability - The applied primer shall be repairable and show no loss of adhesion or other evidence of film failure when tested in accordance with 4.7.17.

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4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

4.1.1 Unless otherwise specified in the contract or purchase order, the manufacturer of the material shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the manufacturer may use his own facilities or any commercial laboratory acceptable to the Aircraft Division. The Aircraft Division reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material conforms to the prescribed requirements.

4.1.2 Inspection records shall be kept complete and available to the Aircraft Division for a period of 3 years. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.2 Classification of Inspection - Inspection requirements are classified as follows:

- a. Qualification
- b. Quality Conformance

4.3 Qualification

4.3.1 Qualification shall require one gallon of base material and the necessary catalyst and thinner (if required), packaged in one quart kits, accompanied by the manufacturer's recommended mixing instructions, and by a test report from an Aircraft Division approved laboratory. The report shall provide evidence of satisfactory compliance to all the requirements of this specification, and shall list by manufacturer's name, formula number, and specification number, the compatible topcoats and sealants.

4.3.2 Qualification shall be based on submittal of a test report in accordance with 4.3.1 and Aircraft Division conducted tests. Aircraft Division conducted tests shall include any tests considered necessary to assure equivalency to previously qualified products.

4.4 Quality Conformance - The following quality conformance tests shall be performed for acceptance of production materials. Each production batch shall be tested using a previously accepted batch of primer, catalyst, and thinner (if specified) as a control. The actual values for nonvolatile, weight per gallon, viscosity, pot life, dry time, and impact resistance shall be reported.

a. Primer Base

Application properties	(4.6.1)
Nonvolatile	(4.7.2)
Weight per gallon	(4.7.1)
Viscosity	(4.7.3)
Pot life	(4.7.4)
Dry time	(4.7.6)
Impact resistance	(4.7.9)
Distilled water	(4.7.11)
Metal anchorage	(4.7.12)
Humidity (7 day exposure)	(4.7.8)
Color	(4.7.7)

b. Primer Catalyst

Nonvolatils	(4.7.2)
Weight per gallon	(4.7.1)

c. Primer Thinner

Nonvolatile	(4.7.2)
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4.4.1 Sampling - Unless otherwise specified, not less than one one-quart kit of primer shall be selected at random from each production batch and subjected to the tests specified in 4.4. Outside contractors see 4.7.21.

4.4.1.1 Batch - A batch shall consist of all material manufactured in one continuous operation and presented for inspection at one time.

4.5 Test Conditions

4.5.1 Standard Conditions - Standard conditions shall be a temperature of $77\text{ F} \pm 2$ and a relative humidity of $50\text{ percent} \pm 5$. All tests shall be conducted at standard conditions unless otherwise specified.

4.5.2 Test Panels

4.5.2.1 All tests shall require 4 specimens of each class for each test condition unless otherwise specified.

4.5.2.2 Description of Test Panels - Test panels shall be identified by the following class designations. Unless otherwise specified, test panels shall be approximately 0.032 by 4 by 6 inches.

- a. Class A - Epoxy fiberglass laminated in accordance with MIL-C-9084 and MIL-R-9300, using style No. 181 fabric.
- b. Class B - Alclad 2024-T3 aluminum alloy (QQ-A-250/5) with immersion chemical treatment in accordance with MIL-C-5541.
- c. Class C - Bare 2024-T3 aluminum alloy (QQ-A-250/4) with immersion chemical treatment in accordance with MIL-C-5541.
- d. Class D - Bare 2024-T3 aluminum alloy (QQ-A-250/4) with chromic acid anodize in accordance with MIL-A-8625, Type I.
- e. Class E - Bare 7178 aluminum alloy (QQ-A-250/14) with chromic acid anodize in accordance with MIL-A-8625, Type I.
- f. Class F - Bare 7075-T6 aluminum alloy (QQ-A-250/12) with sulfuric acid anodize in accordance with MIL-A-8625, Type II.
- g. Class G - Bare 7075-T6 aluminum alloy (QQ-A-250/12) with immersion chemical treatment in accordance with MIL-C-5541.
- h. Class H - AZ31A magnesium alloy (QQ-M-44) with MIL-M-45202, Type I anodic treatment.
- i. Class J - Bare 2024-T3 aluminum alloy (QQ-A-250/12) with sulfuric acid anodize in accordance with MIL-A-8625, Type II. Test panels approximately 0.032 by 12 by 12 inches.
- j. Class K - Bare 2024-T3 aluminum alloy (QQ-A-250/12) with immersion chemical treatment in accordance with MIL-C-5541. Test panels approximately 0.032 by 12 by 12 inches.
- k. Class L - AZ31A magnesium alloy (QQ-M-44) with MIL-M-45202, Type I anodic treatment. Test panels approximately 0.032 by 12 by 12 inches.

4.5.2.3 Cleaning of Test Panels - Immediately prior to application of primer, the surface of each panel shall be cleaned by applying TT-T-266 lacquer thinner directly to the panel to wet the entire surface. While the surface is wet, scrub thoroughly with clean cheesecloth saturated with TT-T-266. Wipe dry with clean cheesecloth before the solvent evaporates. Panels which appear to be of questionable quality for any reason shall be discarded.

4.5.2.4 Handling of Cleaned Panels - Cleaned panels shall be handled only with clean white cotton gloves, shall be stacked clean-side-to-clean-side or individually wrapped in clean chemically neutral paper, and shall be used within 8 hours after cleaning. Panels not used within the 8 hour period shall be recleaned.

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4.6 Preparation of Test Specimens

4.6.1 Mixing and Application of Primer - The primer shall be prepared for spraying by mixing base material, catalyst, and thinner (if required) in the ratio specified by the manufacturer.

4.6.1.1 Apply the primer directly to the applicable panel.

4.6.1.2 Spray a uniformly wet coat to a dry film thickness of 0.5 to 0.7 mil.

4.6.2 Curing of Primer - Unless otherwise specified, all test panels shall be permitted to cure for 168 hours \pm 4 under standard conditions prior to evaluation. The cure of the coating shall be tested in accordance with 4.7.19 on 2 panels of any class.

4.6.3 Application of Sealants

4.6.3.1 Two sets of Class C panels shall be primed in accordance with 4.6.1 and allowed to cure in accordance with 4.6.2. The primed panels shall be cleaned by thoroughly scrubbing with clean cheesecloth wet with naphtha TT-N-95. Panels shall be wiped dry using clean dry cheesecloth. Handle cleaned panels as specified in 4.5.2.4.

4.6.3.2 Coat one set of panels with a continuous layer of properly mixed and qualified MIL-S-8802 sealant and the other with MIL-S-83315 sealant approximately 0.125 inch \pm 0.015 thick on 5 inches of each panel.

4.6.3.3 A 3 by 13 inch strip of cotton duck or cotton drill or a 1 by 12 inch monel screen (cloth or screen with 80 pounds per inch minimum breaking strength) shall be impregnated with the same sealant that is on the panel to which the strip will be applied so that approximately 5 inches on one end is completely covered on both sides. Work sealant well into the cloth or screen.

4.6.3.4 The sealant-impregnated end of the cloth or screen shall be placed on the panel, leaving a loose, unimpregnated end, approximately 7 inches in length. Smooth the cloth or screen down on the layer of sealant, taking care not to trap air under the cloth. An additional 0.125 inch coating of the same sealing material shall be applied over the impregnated cloth.

4.6.3.5 Curing of Sealants - Unless otherwise specified, all sealant coated test panels shall be permitted to cure for 168 hours \pm 4 under standard conditions prior to evaluation.

4.7 Test Methods

4.7.1 Weight Per Gallon - The weight per gallon shall be determined in accordance with Federal Test Method Standard No. 141, Method 4184.

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4.7.2 Nonvolatile Content - The nonvolatile content shall be determined in accordance with Federal Test Method Standard No. 141, Method 4041.1 with the following exceptions:

a. Base Component

1. Sample size shall be 0.75 to 1.25 grams.
2. Add Epon 828 (Shell Chemical Company) plasticizer equal to approximately one half the weight of the sample.

NOTE: The nonvolatile of each batch of Epon 828 shall be determined and the containers shall be kept tightly capped between usage.

3. Thoroughly mix the base and the Epon 828 and bring to a constant weight at 275 F \pm 25.
4. Calculate the percent nonvolatile and correct for percentage Epon 828 nonvolatils.

$$\frac{\text{Weight of sample after heating}}{\text{Weight of sample before heating}} \times 100$$

b. Catalyst - The nonvolatils content of the catalyst shall be determined in accordance with one of the following methods.

1. Federal Test Method Standard No. 141, Method 4041.1.
2. Titration Method for Percent Amine Determination.

a. Reagents

1. Standard HCL solution, approximately 0.5 Normal (N).
2. Bromophenol blue indicator solution.
3. Solvent mixture: 1:1 by volume of ethylene glycol and isopropyl alcohol.

b. Preparation of 0.5N HCL Solution

1. 0.5N HCL solution contains 20.83 milliliters (24.78 grams) of concentrated (12N) HCL solution in 500 ml of solution.
2. Weigh accurately 24.78 grams \pm 0.2 concentrated HCL solution into a 50 ml glass beaker.
3. Transfer to a 500 ml flask with about half its volume filled with deionized water. Wash out beaker with deionized water and transfer to flask. Make up to mark with deionized water, shaking to get a homogeneous mixture.

CAUTION: Never add water to acid.

c. Procedure

1. Sample size is based on percent amine as shown in Table 1.
2. Accurately weigh the specified size sample from Table 1, into a 250 ml Erlenmeyer flask and dilute to 100 ml with solvent mixture. Add 5 drops of bromophenol blue indicator solution and titrate with 0.5N HCL solution until complete disappearance of the blue into a yellowish solution.
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3. Calculate percent amine.

$$\text{Percent Amine} = C \frac{(M \times N)}{(Wt)}$$

Where: M = Milliliters HCL solution used

N = Concentration of HCL solution (normality)

Wt = Weight of the sample

C = Constant which is determined by functionality and molecular weight of the amine or amines in the mixture.

NOTE: Different catalysts have different constants.

TABLE 1. SAMPLE SIZE AMINE DETERMINATION

Percent Amine	Sample Weight, Grams
Up to 5	10 ± 1.0
6 to 11	5 ± 0.5
12 to 17	3 ± 0.5
18 to 27	2 ± 0.2
28 to 55	1 ± 0.1
Above 55	0.5 ± 0.1

c. Thinner (If Required) - The nonvolatile content of the thinner shall be determined in accordance with ASTM D268.

- 4.7.3 Viscosity - The viscosity of the primer mixed with catalyst and thinner (if specified by the manufacturer) shall be determined at 77 F ± 2 one hour after mixing and 8 hours after mixing, using a No. 2 Zahn cup.
- 4.7.4 Pot Life - A one quart container shall be filled to approximately one inch of the top with freshly mixed primer, prepared for spraying in accordance with 4.6.1, and sealed with a tightly fitting cover. The container shall be allowed to remain undisturbed for 16 hours ± 1 under standard conditions. The primer shall be examined for appearance noting skinning, gelling, and seeding. The aged material may be diluted with up to 20 percent (by volume) thinner and shall meet the requirements as specified in 3.3.3.
- 4.7.5 Settling - The settling test shall be conducted in a 100 milliliter (ml) glass-stoppered graduated cylinder. The cylinder shall be filled to the 70 ml mark with freshly mixed primer prepared for spraying in accordance with 4.6.1. The primer cylinder shall be allowed to settle for 16 hours ± 1, -0; then, the cylinder shall be shaken and the length of time required to place all pigment in suspension shall be noted.
- 4.7.6 Dry Time - The dry time shall be determined in accordance with Federal Test Method Standard No. 141, Method 4061, except that Class B panels prepared in accordance with 4.6.1 shall be used.
- 4.7.7 Color - Aluminum faced cardboard panels, 10 caliper paper with 0.00035 inch aluminum foil on one side, shall be coated by spraying to a dry film thickness of 1.0 to 1.5 mils. After 16 hours minimum cure, comparison shall be made with a panel from the last acceptable batch of NAI-1269 primer from the same manufacturer.
- 4.7.8 Humidity Resistance - Class C and Class H panels, prepared and cured in accordance with 4.6.1 and 4.6.2, shall be suspended in a humidity cabinet in accordance with Federal Test Method Standard No. 141, Method 6201. The cabinet shall operate at 120 F ± 2 for 720 to 750 hours with condensing humidity conditions. Panel evaluation after exposure shall include adhesion in accordance with 4.7.18.

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4.7.9 Impact Resistance

- 4.7.9.1 Impact Tester - The impact tester comprises a round-nose steel impact rod, a vertical guide tube, and a base plate. A lift pin attached to the impact rod extends through a slot in the guide tube. A scale along the slot gives foot pounds of impact in steps of 2. To accommodate panels of different thickness, the guide tube is readily raised and lowered. The Gardner Impact Tester shown in Figure 1 meets these requirements.

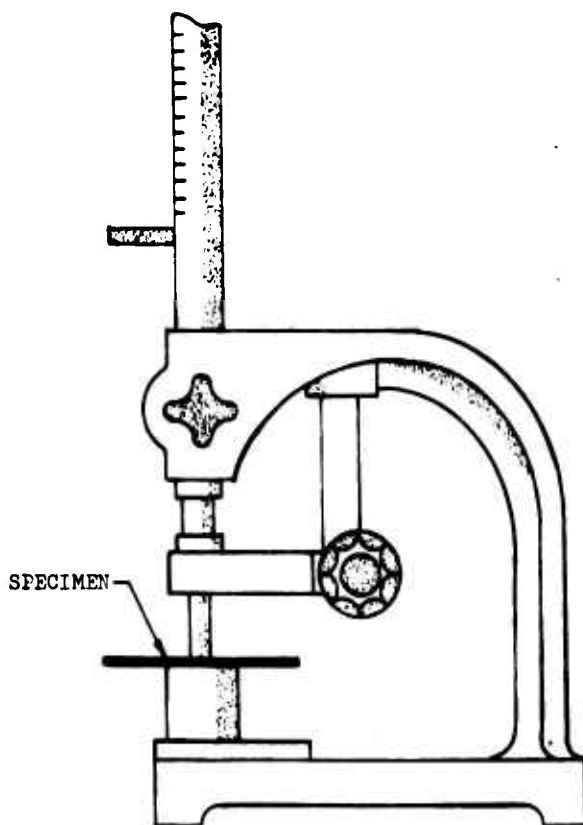


FIGURE 1. GARDNER IMPACT TESTER

- 4.7.9.2 Procedure - Class D panels shall be prepared and cured in accordance with 4.6.1 and 4.6.2 and shall be placed coated side up in a horizontal position in the impact tester. The impact rod shall be raised to 50 inch pounds and released. The uncoated side of the panel shall then be placed up in a horizontal position and the test repeated, raising the impact rod to 30 inch pounds and released. Visually examine the primer film for cracks. Apply masking tape (3M Company No. 250) over the most highly stressed area on the coated side of each impact spot, then remove the tape in one abrupt motion. Loss of adhesion is evidenced by particles of the primer adhering to the tape.

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4.7.10 Corrosion Resistance

4.7.10.1 Class B, D, and H panels shall be primed and cured in accordance with 4.6.1 and 4.6.2. After curing, the panels shall be scribed through the primer and surface treatment to the base metal in 2 diagonal scribe marks extending from corner to corner and tested in 5 percent salt spray in accordance with Federal Test Method Standard No. 141, Method 6061, except that the panels shall be inclined at an angle of approximately 6 degrees from the vertical. Class H panels shall be exposed for 500 to 525 hours. Class B and D panels shall be exposed for 1000 to 1050 hours. Panel evaluation after exposure shall include adhesion in accordance with 4.7.18.

4.7.10.2 Prior to testing, protect backs and edges of panels with corrosion inhibiting primer and seal edges with high melting wax.

4.7.11 Fluid Resistance - Classes of panels as listed below shall be primed and cured in accordance with 4.6.1 and 4.6.2. After curing, the panels shall be tested in accordance with Federal Test Method Standard No. 141, Method 6011, using the fluids and exposure times specified in 3.4.4. Panel evaluation after exposure shall include adhesion in accordance with 4.7.18.

<u>Fluid</u>	<u>Class of Panel Required</u>
Distilled water	A, B, D, E, F, G, H
MIL-H-5606	B, H
MIL-L-7808	B, H

4.7.12 Metal Anchorage - Class B panels, prepared and cured in accordance with 4.6.1 and 4.6.2, shall be tested for anchorage by bending through 180 degrees over a conical mandrel in accordance with Federal Test Method Standard No. 141, Method 6222.

4.7.13 Heat Resistance - Class B panels shall be primed in accordance with 4.6.1 and cured for 24 to 26 hours under standard conditions. After curing, the panels shall be placed in a mechanical convection oven and maintained at $350\text{ F} \pm 10$ for 70 hours ± 1 . The panels shall be removed, cooled to standard conditions, and bent rapidly over a 4 inch diameter mandrel which has been conditioned at the same temperature. Panel evaluation after bending shall include adhesion in accordance with 4.7.18.

4.7.14 Low Temperature Resistance - Class B panels shall be primed and cured in accordance with 4.6.1 and 4.6.2. After curing, the panels shall be placed in a cold box maintained at $-70\text{ F} \pm 5$ for 5 hours. Immediately after removal from the cold box, the panels shall be rapidly bent over a 4 inch diameter mandrel which has been conditioned at the same temperature. Panel evaluation after bending shall include adhesion in accordance with 4.7.18.

4.7.15 Compatibility With Sealants

4.7.15.1 Class C panels shall be primed and cured in accordance with 4.6.1 and 4.6.2 and sealant applied in accordance with 4.6.3. After curing, one panel for each sealant shall be completely immersed for 7 days at $120\text{ F} \pm 2$ in TT-S-735, Type III test fluid. The remaining panels shall be completely immersed in a 3 percent aqueous sodium chloride solution which is covered with a layer of TT-S-735, Type III fluid, for 7 days at $120\text{ F} \pm 2$.

4.7.15.2 Immersion shall be in screw cap jars and the jars shall be sealed by placing a layer of aluminum foil inside the lids.

4.7.15.3 After completion of the immersion period, the jars shall be removed from the heat source and allowed to return to standard temperature prior to removal of specimens. The test shall be completed within 20 hours after removal of the jars from the heat source, and within one hour after removal of the panels from the test fluids.

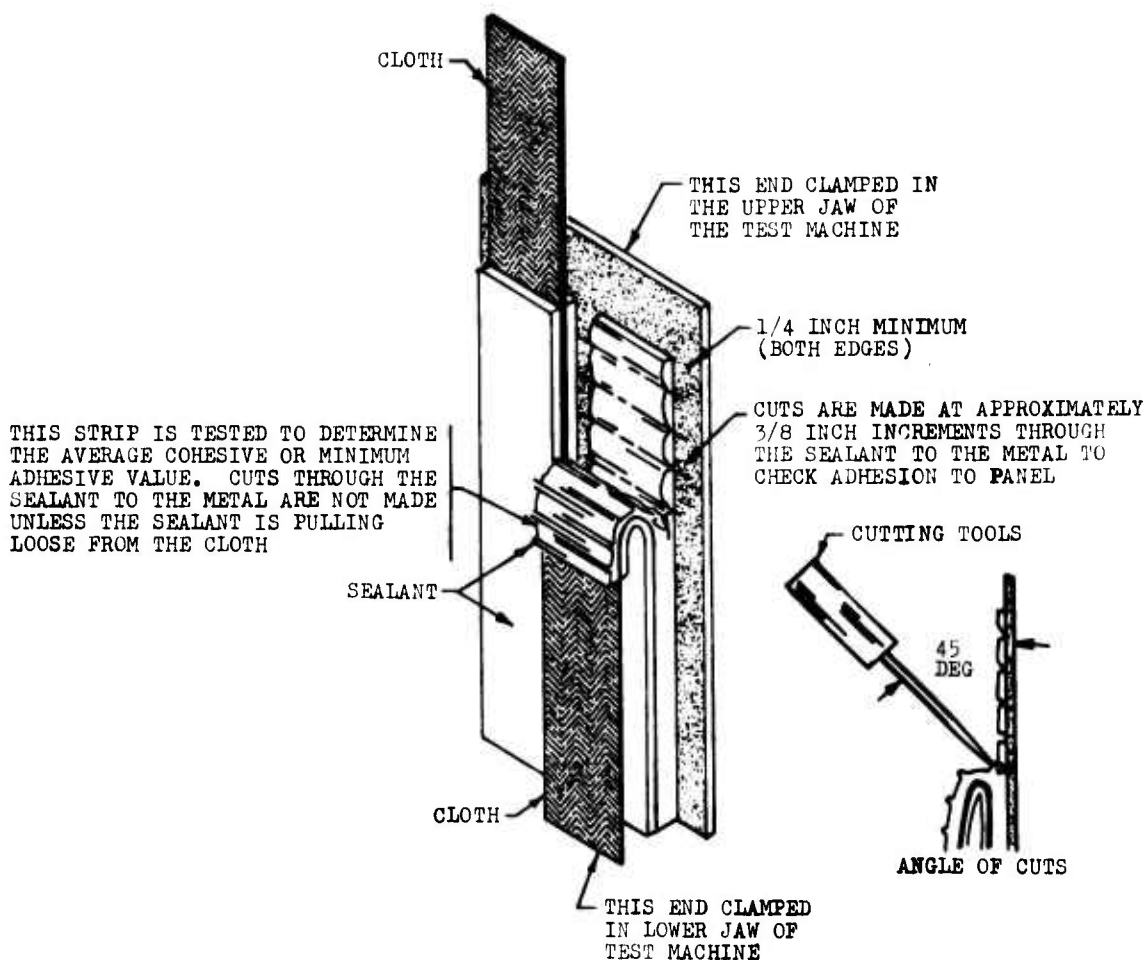
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- 4.7.15.4 After immersion, 2 one inch wide strips shall be prepared through the cloth and sealant to the metal substrate lengthwise along the panel and continuing completely along the unimpregnated cloth. The loose end of each one inch wide strip in turn shall be clamped in one jaw of a suitable recording tensile testing machine and the adjacent end of the panel shall be fastened in the other jaw as shown in Figure 2.
- Cut through the sealant under the cloth, so that an initial separation of the sealant from the panel is promoted.
 - The pull of the cloth shall be at an angle of 180 degrees and a jaw separation rate of 2 inches per minute.



- 4.7.15.5 On one of the strips on the panel, cuts in the sealant to the metal panel at the junction of separation shall be made at an angle of 45 degrees towards the direction of separation at approximately 0.375 inch increments (approximately every 24 seconds) as shown in the diagram. (No cuts are required for 100 percent adhesive failure.) On the other strip, except for the initial cut to promote separation, cuts shall be made only as necessary to prevent the cloth from peeling from the sealant. The percent cohesive failure calculated from the ratio of area of cohesive separation to total area of cohesive and adhesive separation from the metal on both sides of the panel shall be determined. The cohesive strength shall be determined during cohesive tear failure.

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4.7.15.6 The average cohesive strength shall be determined. (Values recorded during cutting shall not be included.)

4.7.16 System Compatibility

4.7.16.1 Class J, K, and L panels shall be cleaned in accordance with 4.5.2.3 and masked so as to divide the panel in 3 equal sections.

4.7.16.2 The 2 outer sections shall be primed with a recently accepted batch of NAI-1269 primer from any other qualified supplier(s). The center section shall be primed with the primer for which qualification is sought. All primers shall be applied in accordance with 4.6.1.

4.7.16.3 Cure 1 to 2 hours under standard conditions and apply a single wet coat of any military specification polyurethane or epoxy topcoat or any Aircraft Division polyurethane topcoat to a dry film thickness of 1 to 1.5 mils.

4.7.16.4 Cure the topcoated panels in accordance with 4.6.2 and subject to the following tests. There shall be no separation of the topcoat from the primed surface.

- a. Metal anchorage (4.7.12)
- b. Impact resistance (4.7.9)
- c. Adhesion (4.7.18)
- d. Humidity (7 day exposure) (4.7.8)

4.7.17 Repairability - Class B and D panels shall be prepared and cured in accordance with 4.6.1 and 4.6.2. After curing, the panels shall be immersed for 168 hours \pm 2 in the following fluids.

- a. Distilled water at 75 F \pm 5
- b. Distilled water at 140 F \pm 2
- c. TT-S-735, Type III fluid at 75 F \pm 2
- d. TT-S-735, Type III fluid at 140 F \pm 2

After exposure, remove, wipe dry with clean dry cheesecloth, then dry 4 hours in an air-circulating oven at 140 F \pm 2. Abrade a small area with 400 grit abrasive sheet to expose the substrate. Clean the panels with TT-T-266 lacquer thinner and brush apply colored chromate chemical treatment (MIL-C-5541) to the exposed substrate. Apply a coat of test primer to the treated area in accordance with 4.6.1 and cure in accordance with 4.6.2. Replace panels in the same test environment for 168 hours \pm 2. Remove panels and test repaired area in accordance with 4.7.18.

4.7.18 Adhesion - Loss of adhesion as a result of other exposure tests shall be determined as follows:

- a. The panels shall be thoroughly dried by wiping with clean dry cheesecloth.
- b. The test shall be conducted at 75 F \pm 2.
- c. Cut 2 parallel scratches one inch apart, through the coating to the metal surface, with a sharp blade knife or scribe. Apply a strip of one inch wide masking tape (3M Company No. 250 not more than 6 months from date of manufacture) across the scratches at 90 degrees to the panel area to be tested. The tape shall be pressed down using 2 passes of a 4.5 pound rubber covered roller approximately 3.5 inches in diameter by 1.75 inches in width. The durometer hardness of the roller surface shall be 70 to 80 on a "D" model.
- d. The tape shall be removed in one abrupt motion, perpendicular to the panels.
- e. Loss of adhesion as evidenced by coating removal or by cracking of the film constitutes failure.

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- 4.7.19 Cure Test - The cure of the dried film in accordance with 4.6.2 shall be tested by saturating a 4 by 4 inch piece of cheesecloth with TT-M-261 methyl ethyl ketone (MEK), flooding the area to be tested with MEK, and using considerable pressure, rubbing the test area 100 times counting only the strokes in one direction. Exposure of any metal constitutes a noncured film.

NOTE: 1. Do not test any area that has been previously wetted with MEK.

2. Some pigment will always be found on the cheesecloth and does not constitute film failure.

- 4.7.20 Physical and Chemical Properties - Control of physical and chemical properties from batch to batch shall be based on qualification values unless otherwise specified. The chemical properties shall be determined by infrared spectrophotometer analysis.

- 4.7.21 Quality Control (Outside Contractors) - Outside contractors using this coating may omit quality control testing provided:

- a. The outside contractor receives written evidence from the manufacturer that a representative sample of the batch, as defined in 4.4.1.1, has been tested and approved by the Aircraft Division Quality Control Laboratory.
- b. For material purchased from the Aircraft Division, each container of the material has been stamped with an Aircraft Division inspection stamp and is dated.

- 4.7.22 Reinspection - All materials shall be reinspected in accordance with 4.4 after one year from date of manufacture, unless all of the batch has been used. If the material passes reinspection test, the usable shelf life shall be extended 6 months. Any materials failing reinspection tests or exceeding the extended shelf life shall be discarded.

4.8 Rejection

- 4.8.1 Any material not conforming to the requirements of this specification shall be rejected.

- 4.8.2 Material rejected in accordance with 4.8.1 may be retested once. For retest, the number of specimens shall be twice the number required by the applicable test method in 4.7. If any retest specimen fails to meet the requirement, the entire batch shall be rejected.

- 4.8.3 No material shall be accepted for delivery that is older than 5 months from date of manufacture.

- 4.9 Reports - Unless otherwise specified, the manufacturer shall furnish with each shipment 3 copies of a report of the results of tests for compliance to the requirements of 4.4 of this specification.

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5. PREPARATION FOR DELIVERY

5.1 Base Material - Each container of base material shall be durably, conspicuously, and legibly marked with the following:

- a. NAI-1269 primer base component (yellow or green)

NOTE: When the manufacturer's label lists other specifications in addition to NAI Specifications, the NAI designation shall be of the same size and type used in listing the other specifications.

- b. Manufacturer's name and formula number
- c. Batch number
- d. Date of manufacture
- e. Purchase order number
- f. Quantity
- g. Caution note as follows:

CAUTION: This base component must be mixed with catalyst (insert appropriate catalyst designation) and thinner (when required - insert appropriate thinner designation), in accordance with applicable Northrop Corporation, Aircraft Division Process Specification prior to use.

5.2 Catalyst - Each container of catalyst shall be durably, conspicuously, and legibly marked with the following:

- a. NAI-1269 Catalyst

NOTE: When the manufacturer's label lists other specifications in addition to NAI Specifications, the NAI designation shall be of the same size and type used in listing the other specifications.

- b. Manufacturer's name and formula number
- c. Batch number
- d. Date of manufacture
- e. Purchase order number
- f. Quantity
- g. Caution note as follows:

CAUTION: This catalyst must be mixed with NAI-1269 base component in accordance with applicable Northrop Corporation, Aircraft Division Process Specification prior to use.

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5.3 Thinner - Each container of thinner shall be durably, conspicuously, and legibly marked with the following:

a. NAI-1269 Thinner

NOTE: When the manufacturer's label lists other specifications in addition to NAI Specifications, the NAI designation shall be of the same size and type used in listing the other specifications.

b. Manufacturer's name and formula number

c. Batch number

d. Date of manufacture

e. Purchase order number

f. Quantity

5.4 Packaging - Packaging shall be such as to assure safe delivery. Each container shall be durably and legibly marked with the following information:

a. NAI-1269 Fluid Resistant Catalyzed Primer

b. Date of manufacture

c. Manufacturer's name

d. Batch number

e. Purchase order number

f. Quantity

g. The unit size as specified on the purchase order

5.4.1 This material shall be available as kits in any size up to 20 gallons composed of the base component plus the required amount of catalyst and thinner (if necessary) or as individual items in any size to 55 gallon drums. The size of a kit is designated by the size of the container of the base component.

5.4.2 The kit size shall be specified on the purchase order.

6. NOTES

6.1 Information pertaining to this specification may be obtained from Materials Engineering (3495/32), Aircraft Division.

6.2 Suppliers may obtain information pertaining to, or additional copies of, this specification from Northrop Corporation, Aircraft Division, Materiel Department (6000/71), 2031 E. Mariposa Ave., El Segundo, California 90245.

RESPONSIBLE ENGINEER <i>J. P. Kinsling</i> AIRCRAFT DIVISION SPECIFICATION CONTROL PROJECT OFFICE	NORTHROP Northrop Corporation Aircraft Division QUALIFIED PRODUCTS LIST	NOR-QPL-1269 DATE 2 May 1972 RELEASE EO D56942 CODE IDENT. NO 76823
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TITLE:

FLUID RESISTANT CATALYZED PRIMER

(This revision supersedes NOR-QPL-1269, dated 5-28-71.)

PRODUCTS QUALIFIED UNDER SPECIFICATION NAI-1269

Manufacturer's Designation	Manufacturer's Name and Address	Qualification	
		Code Number	Date
Brolite Fluid Resistant Catalyzed Primer Base P-543-D-66 Converter T-1159-66	Andrew Brown Company Los Angeles, California	2	8-3-70
	Nonvolatile 47.0 Weight per Gallon 9.3		
	Method 1 Nonvolatile 9.1 Weight per Gallon 7.2		
Cat-A-Lac Primer Base 463-6-5 Catalyst X-306 Reducer TL-65	Finch Paint & Chemical Co. Torrance, California	4	12-7-64
	Nonvolatile 47.9 Weight per Gallon 9.7		
	Method 1 Nonvolatile 5.5 Weight per Gallon 6.9		
	Method 2 Percent Amine NV 11.30 Constant "C" 10.70 Weight per Gallon 6.9		
	Weight per Gallon 7.15	3	5-15-62
Cat-A-Lac Primer Base 463-6-12 Rule 66 Catalyst X-315 Reducer TL-65	Finch Paint & Chemical Co. Torrance, California	3	4-19-68
	Nonvolatile 47.9 Weight per Gallon 9.7		
	Method 1 Nonvolatile 5.5 Weight per Gallon 6.9		
	Method 2 Percent Amine N-V 11.56 Constant "C" 10.50 Weight per Gallon 6.9	3	5-15-68
	Weight per Gallon 7.15		
Cat-A-Lac Primer Base 463-6-11 Rule 66 Catalyst X-315 Reducer TL-65	Finch Paint & Chemical Co. Torrance, California	3	4-19-68
	Nonvolatile 47.9 Weight per Gallon 9.7		
	Method 1 Nonvolatile 5.5 Weight per Gallon 6.9		
	Method 2 Percent Amine N-V 11.56 Constant "C" 10.50 Weight per Gallon 6.9	3	5-15-68
	Weight per Gallon 7.15		

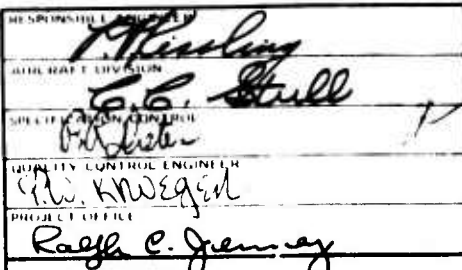
NORTHROPNorthrop Corporation
Aircraft Division

DATE 2 May 1972

PRODUCTS QUALIFIED UNDER SPECIFICATION NAI-1269 (CONTINUED)

Manufacturer's Designation	Manufacturer's Name and Address	Qualification	
		Code Number	Date
FR Epoxy Primer LXP-5R-3	Deft, Inc., Torrance, California	3	6-23-67
Base	Nonvolatile 43.5 Weight per Gallon 9.1		
Catalyst	Nonvolatile 4.2 Weight per Gallon 7.1		
Reducer IS 108B	Weight per Gallon 7.66		
FR Epoxy Primer LXP-16A (Aerosol)	Deft, Inc., Torrance, California	3	5-4-70 See Note 1
Base	Nonvolatile 59.4 Weight per Gallon 10.1		
Catalyst	Nonvolatile 1.0 Weight per Gallon 7.0		
Fluid Resistant Catalyzed Primer	Fuller-O'Brien Corp., S. San Francisco, California	2	1-5-70
Base 162-Y-47	Nonvolatile 59.0 Weight per Gallon 10.7		
Catalyst 75-X-35	Nonvolatile 4.9 Weight per Gallon 6.9		
Super Koropon	DeSoto Chemical Coatings, Inc., Berkeley, California	3	4-19-68
Primer Base 513-700	Nonvolatile 61.9 Weight per Gallon 10.2		
Catalyst 910-707	Method 1 Nonvolatile 5.8 Weight per Gallon 7.0		
	Method 2 Percent Amine N-V 6.0 Constant "C" 10.2 Weight per Gallon 7.0		
Super Koropon	DeSoto Chemical Coatings, Inc., Berkeley, California	4	4-11-62
Primer Base 515-003	Nonvolatile 62.0 Weight per Gallon 10.9		
Catalyst 910-012	Method 1 Nonvolatile 3.6 Weight per Gallon 6.9		
	Method 2 Percent Amine N-V 5.8 Constant "C" 10.2 Weight per Gallon 6.9		

NOTE: 1. Supplier shall supply production samples as specified in paragraph 4.4.1 for approval by Aircraft Division Quality Control prior to aerosol packaging.

<div style="text-align: center;">  </div>	NORTHROP Northrop Corporation Aircraft Division MATERIAL SPECIFICATION	<div style="text-align: right; font-size: 2em; font-weight: bold;">NAI-1330</div> <table style="width: 100%; border: none;"> <tr> <td style="border: none;"> DATE 1 October 1975 RELEASE EO E07194 CODE IDENT NO 76823 </td> <td style="width: 10%; text-align: center; vertical-align: middle; border: 1px solid black;"> <div style="font-size: 3em; font-weight: bold;">A</div> <div style="font-weight: bold;">REVISION</div> </td> </tr> </table>	DATE 1 October 1975 RELEASE EO E07194 CODE IDENT NO 76823	<div style="font-size: 3em; font-weight: bold;">A</div> <div style="font-weight: bold;">REVISION</div>																														
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TITLE: ELECTRODEPOSITED CORROSION INHIBITING, FLUID RESISTANT PRIMER																																		
<p>(This revision supersedes Material Specification NAI-1330, dated 29 July 1969. Changes in this revision are not identified.)</p> <p>1. <u>SCOPE</u></p> <p>1.1 This specification establishes the requirements for an electrodeposited, corrosion inhibiting, fluid resistant primer to be used alone or in conjunction with a topcoat.</p> <p>1.2 The primer, as controlled by this specification, is intended for interior or exterior use on aerospace vehicles. It shall be used as a corrosion inhibiting coating or as a corrosion inhibiting undercoat for lacquers, enamels, epoxies, or polyurethane topcoats.</p> <p>1.3 Primers meeting the requirements of this specification meet the requirements of the Los Angeles County Air Pollution Control District, Rule 66, for materials to be used within Los Angeles County, California.</p> <p>2. <u>APPLICABLE DOCUMENTS</u></p> <p>2.1 The following publications of the issue in effect on the date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.</p> <p>2.2 <u>Government</u></p> <p>2.2.1 <u>Specifications</u></p> <table style="width: 100%; border: none;"> <tr> <td style="width: 40%;">2.2.1.1 O-A-51</td> <td>Acetone, Technical</td> </tr> <tr> <td>2.2.1.2 QQ-A-250/4</td> <td>Aluminum Alloy 2024, Plate and Sheet</td> </tr> <tr> <td>2.2.1.3 QQ-A-250/5</td> <td>Aluminum Alloy Alclad 2024, Plate and Sheet</td> </tr> <tr> <td>2.2.1.4 QQ-A-250/12</td> <td>Aluminum Alloy 7075, Plate and Sheet</td> </tr> <tr> <td>2.2.1.5 QQ-M-44</td> <td>Magnesium Alloy Plate and Sheet (AZ31B)</td> </tr> <tr> <td>2.2.1.6 TT-L-32</td> <td>Lacquer, Cellulose-Nitrate, Gloss, For Aircraft Use</td> </tr> <tr> <td>2.2.1.7 TT-M-261</td> <td>Methyl Ethyl Ketone, Technical</td> </tr> <tr> <td>2.2.1.8 TT-N-95</td> <td>Naptha, Aliphatic</td> </tr> <tr> <td>2.2.1.9 TT-S-735</td> <td>Standard Test Fluids; Hydrocarbon</td> </tr> <tr> <td>2.2.1.10 TT-T-266</td> <td>Thinner, Lacquer</td> </tr> <tr> <td>2.2.1.11 CCC-C-419</td> <td>Cloth, Duck Cotton, Unbleached, Plied Yarns Army and Numbered</td> </tr> <tr> <td>2.2.1.12 MIL-C-5541</td> <td>Chemical Conversion Coatings on Aluminum and Aluminum Alloys</td> </tr> <tr> <td>2.2.1.13 MIL-H-5606</td> <td>Hydraulic Fluid, Petroleum Base; Aircraft Missile, and Ordnance</td> </tr> <tr> <td>2.2.1.14 MIL-V-5624</td> <td>Turbine Fuel, Aviation, Grades JP-4 and JP-5.</td> </tr> <tr> <td>2.2.1.15 MIL-L-7808</td> <td>Lubricating Oil, Aircraft Turbine Engine, Synthetic Base</td> </tr> <tr> <td>2.2.1.16 MIL-S-8802</td> <td>Sealing Compound, Temperature-Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High Adhesion</td> </tr> </table>			2.2.1.1 O-A-51	Acetone, Technical	2.2.1.2 QQ-A-250/4	Aluminum Alloy 2024, Plate and Sheet	2.2.1.3 QQ-A-250/5	Aluminum Alloy Alclad 2024, Plate and Sheet	2.2.1.4 QQ-A-250/12	Aluminum Alloy 7075, Plate and Sheet	2.2.1.5 QQ-M-44	Magnesium Alloy Plate and Sheet (AZ31B)	2.2.1.6 TT-L-32	Lacquer, Cellulose-Nitrate, Gloss, For Aircraft Use	2.2.1.7 TT-M-261	Methyl Ethyl Ketone, Technical	2.2.1.8 TT-N-95	Naptha, Aliphatic	2.2.1.9 TT-S-735	Standard Test Fluids; Hydrocarbon	2.2.1.10 TT-T-266	Thinner, Lacquer	2.2.1.11 CCC-C-419	Cloth, Duck Cotton, Unbleached, Plied Yarns Army and Numbered	2.2.1.12 MIL-C-5541	Chemical Conversion Coatings on Aluminum and Aluminum Alloys	2.2.1.13 MIL-H-5606	Hydraulic Fluid, Petroleum Base; Aircraft Missile, and Ordnance	2.2.1.14 MIL-V-5624	Turbine Fuel, Aviation, Grades JP-4 and JP-5.	2.2.1.15 MIL-L-7808	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base	2.2.1.16 MIL-S-8802	Sealing Compound, Temperature-Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High Adhesion
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Northrop Corporation
Aircraft Division

MATERIAL SPECIFICATION: NAI-1330
Revision A

DATE 1 October 1975

- | | |
|--|--|
| 2.2.1.17 MIL-C-22750 | Coating, Epoxy Polyamide |
| 2.2.1.18 MIL-C-22751 | Coating Systems, Epoxy-Polyamide, Chemical and Solvent Resistant: Process for Application of |
| 2.2.1.19 MIL-P-23377 | Primer Coating, Epoxy-Polyamide, Chemical and Solvent Resistant |
| 2.2.1.20 MIL-L-23699 | Lubricating Oil, Aircraft Turbine Engines, Synthetic Base |
| 2.2.1.21 MIL-C-81706 | Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys |
| 2.2.1.22 MIL-S-81733 | Sealing and Coating Compound, Corrosion Inhibited |
| 2.2.1.23 MIL-S-83315 | Sealing Compound, Aluminum Structure, Pressure and Weather Sealing, Low Density |
| 2.2.2 <u>Standards</u> | |
| 2.2.2.1 Federal Test Method Standard No. 141 | Paints, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling and Testing |
| 2.2.2.2 Federal Standard No. 595 | Colors |
| 2.3 <u>Aircraft Division</u> | |
| 2.3.1 C-30 | Cleaning After Forming-Magnesium |
| 2.3.2 FP-80 | Application of Fluid Resistant Epoxy Primer |
| 2.3.3 NAI-1269 | Fluid Resistant Catalyzed Primer |
| 2.3.4 NAI-1278 | Coating, Gloss Polyurethane Topcoat, for Aircraft Use |
| 2.3.5 NAI-1290 | Coating, Camouflage Polyurethane Topcoat, for Aircraft Use |
| 3. <u>REQUIREMENTS</u> | |
| 3.1 <u>Qualifications</u> | |
| 3.1.1 The primer supplied to this specification shall be water reducible. | |
| 3.1.2 <u>Qualification Prior to Production</u> | |
| 3.1.2.1 This specification requires products qualification prior to acceptance or production orders. All materials furnished by a supplier to this specification shall meet all requirements listed herein, regardless of the extent of quality control tests. | |
| 3.1.2.2 Materials submitted for qualification shall be accompanied by a test report from an Aircraft Division approved laboratory. The report shall provide evidence of satisfactory compliance to the requirements of this specification, and shall list by manufacturer's name, formula number, and specification number, the compatible topcoats and sealants. A list of currently used primers, topcoats, and sealants may be obtained from Aircraft Division Materials Engineering. | |
| 3.1.2.3 Product qualification is granted only by Aircraft Division Materials Engineering. | |
| 3.1.2.4 No change in the qualitative values of the tank charge material or method of manufacture shall be made subsequent to qualification without notification and prior approval in writing from Aircraft Division Materials Engineering. Requalification of a revised tank charge material or method of manufacture shall be required. | |
| 3.1.2.5 The feed or make-up formula (material) may vary as designated by the manufacturer so as to maintain the operating tank (bath) within the range specified in the applicable process specification. | |
| 3.1.2.6 After qualification, all subsequent shipment shall be from the same manufacturing plant that submitted the approval qualification sample. Any other plant, although from the same company, shall be approved before material is shipped from that plant. | |

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3.2 Materials

- 3.2.1 Vehicle - The vehicle used in the formulation of this primer shall be heat curable at a temperature of $275\text{ F} \pm 5$ ($135\text{ C} \pm 3$) when tested in accordance with 4.6.3.
- 3.2.2 Application - The material covered by this specification is to be mixed with water as specified by the manufacturer to produce a suitable viscosity for application by electro-deposition.
- 3.2.3 Appearance - The material shall be free of skins, lumps, grit, and all foreign contaminants and shall be easily mixed to a smooth homogeneous condition as received and after one year of inside storage at 50 F (10 C) or below.
- 3.2.4 Odor - The odor of the primer shall not cause discomfort to operator applying the materials.
- 3.2.5 Toxicity - Materials meeting the requirements of this specification shall have no adverse effect on the health of personnel during use or curing when used for the intended purpose.

3.3 Physical Properties

3.3.1 Weight Per Gallon

- 3.3.1.1 The weight per gallon of the basic tank charge and the makeup material shall not vary by more than ± 0.2 pound per gallon when tested in accordance with 4.7.1. The established value for the basic tank charge material shall be 9.5 to 10.5 pounds per gallon. The weight per gallon of the feed or makeup formula (material) may vary as designated by the manufacturer so as to maintain the operating tank (bath) within the range specified in the applicable process specification.
- 3.3.1.2 The actual values established during qualification shall be listed on the Qualified Products List for the basic tank charge material.

3.3.2 Nonvolatile Content

- 3.3.2.1 The nonvolatile content of the primer shall not vary from the established percentage by more than ± 2 percent.
- 3.3.2.2 The actual values established during qualification of each material shall be listed on the Qualified Products List.

3.3.3 Viscosity

- 3.3.3.1 The as received viscosity of the basic tank charge shall be 100 to 500 Krebs Units (KU's) when tested in accordance with 4.7.4.
- 3.3.3.2 The as received viscosity of the feed or makeup formula (material) may vary as designated by the manufacturer so as to maintain the operating tank (bath) within the range specified in the applicable process specification.

3.3.4 Tank Life

- 3.3.4.1 There shall be no evidence of skinning, gelling, or seeding of the primer in the operating tank after the tank has been allowed to stand unagitated at room temperature for up to 72 hours when the primer is applied in accordance with 4.6.2. If necessary, the tank may be purged by ultrafiltration and the solid restored to the solids range established on qualification by the addition of not more than 20 percent (by volume) of new material. The restored material shall meet the established solids requirements. Tanks shall have a minimum working life of one year with at least two turnovers of feed or makeup material per year.
- 3.3.4.2 When applied in accordance with 3.3.4.1, the resulting coating shall have satisfactory surface appearance and shall meet the following requirements of this specification.
 - a. Surface appearance - 3.2.3
 - b. Cure time - 3.3.6
 - c. Cure temperature - 3.3.6
 - d. Color - 3.3.7
 - e. Humidity - 3.4.1
 - f. Fluid immersion;
 - 1. MIL-L-7808 jet engine oil - 3.4.4.1c
 - 2. Distilled water - 3.4.4.1a
 - g. Metal anchorage - 3.4.5

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- 3.3.5 Settling - The solids content of the ready-to-use tank shall be completely redispersed and suitable for application upon shaking after having been permitted to stand undisturbed for 72 hours when tested in accordance with 4.7.3.
- 3.3.6 Cure Time - The applied coating shall be fully cured after baking 35 to 45 minutes at a temperature of $275\text{ F} \pm 5$ ($135\text{ C} \pm 3$) when tested in accordance with 4.7.5.
- 3.3.7 Color - The color of the primer shall be yellow-green, approximating Federal Standard No. 595, Color 14533, characteristic of the pigments used.
- 3.4 Film Properties
- 3.4.1 Humidity Resistance - The primer shall show no blistering, loss of adhesion, or removal with the aircraft fluids listed in 3.4.4.1, after 3000 hours in condensing humidity at $120\text{ F} \pm 2$ ($49\text{ C} \pm 1$) when tested in accordance with 4.7.7.
- 3.4.2 Impact Resistance - The primer shall not crack or lose adhesion on either surface when subjected to an impact of 80 inch pounds and tested in accordance with 4.7.10.
- 3.4.3 Corrosion Resistance - The applied primer shall protect the coated substrate from corrosion for a minimum of 2500 hours when exposed to 5 percent salt spray and tested in accordance with 4.7.11. Corrosion within 0.125 inch of the scribe mark shall not be considered failure.
- 3.4.4 Fluid Resistance
- 3.4.4.1 Room Temperature - The applied primer shall withstand immersion in the following fluids at room temperature $75\text{ F} \pm 5$ ($23\text{ C} \pm 3$) for the time specified without blistering, loss of adhesion, softening, or other film failures when tested in accordance with 4.7.13.1. Blistering which is obviously caused by contamination, such as fingerprints or marring of the uncured primer, shall not be cause for rejection.
- | | |
|------------------------------|----------|
| a. Distilled water | 150 days |
| b. MIL-H-5606 fluid | 150 days |
| c. MIL-L-7808 jet engine oil | 150 days |
| d. MIL-L-23699 oil | 150 days |
| e. MIL-V-5624 JP-5 jet fuel | 150 days |
| f. Skydrol 500 | 150 days |
- 3.4.4.2 Elevated Temperature - The applied primer shall withstand immersion in the following fluids at $250\text{ F} \pm 5$ ($121\text{ C} \pm 3$) for the time specified without blistering, loss of adhesion, softening, or other film failures when tested in accordance with 4.7.13.2. Blistering which is obviously caused by contamination, such as fingerprints or marring of the uncured primer, shall not be cause for rejection.
- | | |
|------------------------------|---------------------------|
| a. MIL-L-7808 jet engine oil | 24 hours at 250 F (121 C) |
| b. MIL-L-23699 oil | 24 hours at 250 F (121 C) |
- 3.4.5 Metal Anchorage - The applied primer shall show satisfactory adhesion to chemically treated metal with no flaking and no cracking when tested on a conical mandrel in accordance with 4.7.9.
- 3.4.6 Heat Resistance - The applied primer shall be heat resistant to 300 F (149 C) long term and 350 F short term and shall show no blistering, loss of adhesion, or other evidence of film failure when tested in accordance with 4.7.12. Discoloration or cracking on both without adhesion loss shall not be cause for rejection.
- 3.4.7 Low Temperature Resistance - The applied primer shall have low temperature resistance to -70 F (-57 C) and shall show no loss of adhesion or other evidence of film failure when tested in accordance with 4.7.14.
- 3.4.8 Sealant Compatibility
- 3.4.8.1 The applied primer shall be compatible with MIL-S-8802, MIL-S-83315, and MIL-S-81733 sealing compound.

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- 3.4.8.2 When tested for compatibility with sealants, the peel strength of the sealant shall average at least 20 pounds per inch peel with no value lower than 15 pounds for any one specimen. The separation shall be a minimum of 95 percent in cohesion of the sealant, disregarding areas of sealant to cloth failures, when tested in accordance with 4.7.16.
- 3.4.9 Finish System Compatibility - The applied primer from any qualified supplier shall be compatible with NAI-1269 epoxy primer, NAI-1278 gloss polyurethane, and NAI-1290 camouflage polyurethane topcoat of any qualified supplier and all government specification topcoats specified by the contracting officer when applied as part of a finish system and tested in accordance with 4.7.17.
- 3.4.10 Repairability - The applied primer shall be repairable and show no loss of adhesion or other evidence of film failure when repaired with NAI-1269 or MIL-P-23377 epoxy primer and tested in accordance with 4.7.15.
- 3.5 Chemical Properties - The chemical properties of each approved primer shall be within the range shown on the infrared curve for each primer on the Qualified Products List.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

- 4.1.1 Unless otherwise specified in the contract or purchase order, the manufacturer of the material shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the manufacturer may use his own facilities or any commercial laboratory acceptable to the Aircraft Division. The Aircraft Division reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material conforms to the prescribed requirements.
- 4.1.2 Inspection records shall be kept complete and available to the Aircraft Division in accordance with contract or purchase order. These records shall contain all data necessary to determine compliance with the requirements of this specification.

4.2 Classification of Inspection - Inspection requirements are classified as follows:

- a. Qualification
- b. Quality Conformance

4.3 Qualification

- 4.3.1 Qualification shall require ten gallons of tank charge material packaged in one gallon cans, accompanied by the manufacturer's recommended mixing instructions, and by a test report from an Aircraft Division approved laboratory. The report shall provide evidence of satisfactory compliance to all the requirements of this specification, and shall list by manufacturer's name, formula number, and specification number, the compatible primers, topcoats, and sealants.
- 4.3.2 Qualification shall be based on submittal of a test report in accordance with 4.3.1 and Aircraft Division conducted tests. Aircraft Division conducted tests shall include any tests considered necessary to assure equivalency to previously qualified products.

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- 4.4 Quality Conformance - The following quality conformance tests shall be performed for acceptance of production materials. Each production batch shall be tested using a previously accepted batch of primer as control. The actual values for nonvolatile, weight per gallon, viscosity, settling, cure time, cure temperature, and impact resistance shall be reported.

Appearance properties	3.2.3
Nonvolatile	4.7.2
Weight per gallon	4.7.1
Viscosity	4.7.4
Color	4.7.6
Cure time	4.6.3
Cure temperature	4.6.3
Impact resistance	4.7.10
Humidity (14 day exposure)	4.7.7
Sealant compatibility	
a. MIL-S-8802	4.7.16
b. MIL-S-81733	4.7.16
c. MIL-S-83315	4.7.16
Metal anchorage	4.7.9
Fluid resistance (14 day exposure)	
a. MIL-L-7808	4.7.13
b. Skydrol 500	4.7.13.1
c. Distilled water	4.7.13

4.4.1 Sampling

- 4.4.1.1 Unless otherwise specified, not less than two gallons of primer shall be drawn at random from each production batch and subjected to the tests specified in 4.4.
- 4.4.1.2 Batch - A batch shall consist of all material of one manufacturing control number and presented for inspection at one time.

4.5 Test Conditions

- 4.5.1 Standard Conditions - Standard conditions shall be a temperature of $77\text{ F} \pm 2$ ($25\text{ C} \pm 1$) and a relative humidity of 50 percent ± 5 . All tests shall be conducted at standard conditions unless otherwise specified.

4.5.2 Test Panels

- 4.5.2.1 All tests shall require 4 specimens of each class for each test condition unless otherwise specified.

- 4.5.2.2 Description of Test Panels - Test panels shall be identified by the following class designations. Unless otherwise specified, test panels shall be approximately 0.032 by 4 by 6 inches.

- Class A - Alclad 2024-T3 aluminum alloy (QQ-A-250/5) with immersion chemical treatment in accordance with MIL-C-5541/MIL-C-81706.
- Class B - Bare 2024-T3 aluminum alloy (QQ-A-250/4) with immersion chemical treatment in accordance with MIL-C-5541/MIL-C-81706.
- Class C - Bare 7075-T6 aluminum alloy (QQ-A-250/12) with immersion chemical treatment in accordance with MIL-C-5541.
- Class D - Bare 7075-0 aluminum alloy (QQ-A-250/12) with immersion chemical treatment in accordance with MIL-C-5541.
- Class E - AZ31A magnesium alloy (QQ-M-44) without surface treatment.

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4.5.2.3 Cleaning and Preparation of Test Panels

- a. Aluminum Alloys - Aluminum alloy test panels shall be cleaned and processed in accordance with MIL-C-5541.
- b. Magnesium Alloys - Magnesium alloy test panels shall be cleaned in accordance with Process Specification C-30, Method 1 prior to coating.
- c. After cleaning, panels which appear to be of questionable quality for any reason shall be discarded.

4.5.2.4 Handling of Cleaned Panels - Cleaned and surface treated panels shall be handled only with clean white cotton gloves, and if not immediately used, wrapped in clean chemically neutral paper. Panels shall be used within 48 hours after cleaning or surface treatment. Panels not used within 48 hours shall be recleaned.

4.6 Preparation of Test Specimens

4.6.1 Mixing of Primer - Primer shall be prepared for application by mixing and thinning as specified by the manufacturer. After mixing, allow the primer to stabilize as specified by the manufacturer before using.

4.6.2 Application of Primer

4.6.2.1 Cleaned or clean surface treated panels shall be thoroughly wet with deionized water when immersed in the primer.

4.6.2.2 Apply primer by electrodeposition to a uniform dry film thickness of 0.2 to 0.3 mil.

NOTE: Tank shall have been under agitation at least 4 hours before primer application.

4.6.3 Curing of Primer - Unless otherwise specified, all test panels shall be cured in a constant temperature oven at $275\text{ F} \pm 5$ ($135\text{ C} \pm 3$) for 35 to 45 minutes prior to evaluation. The cured coating shall be tested in accordance with 4.7.5 on 2 panels of any class.

4.6.4 Application of Sealants

4.6.4.1 Three sets of Class B panels shall be primed in accordance with 4.6.2 and cured in accordance with 4.6.3. The primed panels shall be cleaned by thoroughly scrubbing with clean cheesecloth wet with naptha TT-N-95. Panels shall be wiped dry using clean dry cheesecloth. Handle cleaned panels as specified in 4.5.2.4.

4.6.4.2 Coat one set of panels with a continuous layer of properly mixed and qualified MIL-S-8802 sealant, one set with MIL-S-81733, and the other with MIL-S-83315 sealant approximately 0.125 inch \pm 0.015 thick on 5 inches of each panel.

4.6.4.3 A 3 by 13 inch strip of cotton duck or cotton drill or a 1 by 12 inch monel screen (cloth or screen with 80 pounds per inch minimum breaking strength) shall be impregnated with the same sealant that is on the panel to which the strip will be applied so that approximately 5 inches on one end is completely covered on both sides. Work sealant well into the cloth or screen.

4.6.4.4 The sealant-impregnated end of the cloth or screen shall be placed on the panel, leaving a loose, unimpregnated end, approximately 7 inches in length. Smooth the cloth or screen down on the layer of sealant, taking care not to trap air under the cloth. An additional 0.125 inch coating of the same sealing material shall be applied over the impregnated cloth.

4.6.4.5 Curing of Sealants - Unless otherwise specified, all sealant coated test panels shall be permitted to cure for 168 hours \pm 4 under standard conditions prior to evaluation.

4.7 Test Methods

4.7.1 Weight Per Gallon - The weight per gallon shall be determined in accordance with Federal Test Method Standard No. 141, Method 4184.

4.7.2 Nonvolatile - The nonvolatile content of the primer shall be determined in accordance with Federal Test Method Standard No. 141, Method 4041.

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4.7.3 Settling - The settling test shall be conducted in a 100 milliliter (ml) glass-stoppered graduated cylinder. The cylinder shall be filled to the 70 ml mark with freshly mixed primer prepared in accordance with 4.6.1. The filled cylinder shall be allowed to settle for 16, 40, and 72 hours, each period shall be ± 1 hour. At the conclusion of each period, the cylinder shall be shaken and the length of time required to place all pigment in suspension shall be noted.

4.7.4 Viscosity - The viscosity of the primer shall be determined in accordance with Federal Test Method Standard No. 141, Method 4281.

4.7.5 Cure Test - The cure of the dried film in accordance with 4.6.3 shall be tested by saturating a 4 by 4 inch piece of cheesecloth with TT-M-261 methyl ethyl ketone (MEK), flooding the area to be tested with MEK, and using considerable pressure, rubbing the test area 100 times counting only the strokes in one direction. Exposure of any metal constitutes a noncured film.

NOTES: 1. Do not test any area that has been previously wetted with MEK.

2. Some pigment will always be found on the cheesecloth and does not constitute film failure.

4.7.6 Color - One set of Class B panels shall be primed in accordance with 4.6.2 and cured in accordance with 4.6.3. After curing, comparison shall be made with a panel from the last acceptable batch of NAI-1330 primer from the same manufacturer.

4.7.7 Humidity Resistance - Class A, Class C, and Class D panels, prepared and cured in accordance with 4.6.2 and 4.6.3, shall be suspended in a humidity cabinet in accordance with Federal Test Method Standard No. 141, Method 6201. The cabinet shall operate at $120\text{ F} \pm 2$ ($49\text{ C} \pm 1$) with condensing humidity conditions. Class A and C panels shall be exposed for $3000\text{ hours} \pm 50$. Class D panels shall be exposed for $1000\text{ hours} \pm 25$. Panel evaluation after exposure shall include adhesion in accordance with 4.7.8.

4.7.8 Adhesion - Loss of adhesion as a result of other exposure tests shall be determined as follows:

a. The panels shall be thoroughly dried by wiping with clean dry cheesecloth.

b. The test shall be conducted at $75\text{ F} \pm 2$ ($23\text{ C} \pm 1$).

c. Cut 2 parallel scratches one inch apart, through the coating to the metal surface, with a sharp blade knife or scribe. Apply a strip of one inch wide masking tape (3M Company No. 250 not more than 6 months from date of manufacture) across the scratches at 90 degrees to the panel area to be tested. The tape shall be pressed down using 2 passes of a 4.5 pound rubber covered roller approximately 3.5 inches in diameter by 1.75 inches in width. The durometer hardness of the roller surface shall be 70 to 80 on a "D" model.

d. The tape shall be removed in one abrupt motion, perpendicular to the panels.

e. Loss of adhesion as evidenced by coating removal or by cracking of the film constitutes failure.

4.7.9 Metal Anchorage - Any class of panels shall be prepared and cured in accordance with 4.6.2 and 4.6.3. After aging at room temperature a minimum of 16 hours, the anchorage of the coating shall be determined by bending the panel through 180 degrees over a conical mandrel in accordance with Federal Test Method Standard No. 141, Method 6222.

4.7.10 Impact Resistance

4.7.10.1 Impact Tester - The impact tester comprises a round-nose steel impact rod, a vertical guide tube, and a base plate. A lift pin attached to the impact rod extends through a slot in the guide tube. A scale along the slot gives foot pounds of impact in steps of 2. To accommodate panels of different thickness, the guide tube is readily raised and lowered. The Gardner Impact Tester shown in Figure 1 meets these requirements.

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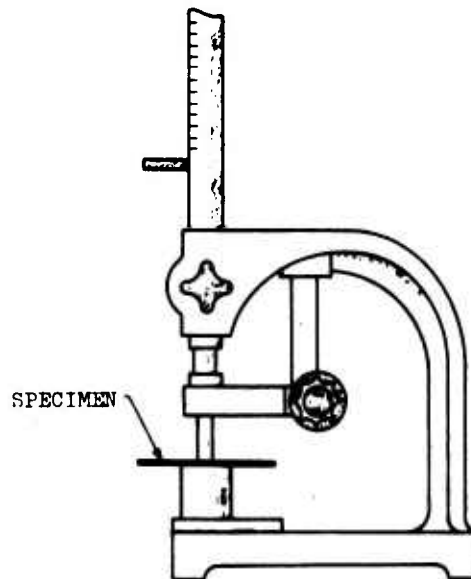


FIGURE 1. GARDNER IMPACT TESTER

- 4.7.10.2 **Procedure** - Class D panels shall be prepared and cured in accordance with 4.6.2 and 4.6.3 and shall be placed in a horizontal position in the impact tester. The impact rod shall be raised to 80 inch-pounds and released. The panel shall be removed from the tester and turned so that the impacted side is facing down and reinserted in the tester. The impact rod shall again be raised to 80 inch-pounds and released. Visually examine the primer for cracks. Apply masking tape (3M Company No. 250) over the most highly stressed area of each impact spot using maximum thumb pressure. Remove tape in one abrupt motion. Loss of adhesion is evidenced by particles of the primer adhering to the tape.
- 4.7.11 **Corrosion Resistance** - Class A, C, and E panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be scribed through the primer and surface treatment to the base metal in 2 diagonal scribe marks extending from corner to corner and tested in 5 percent salt spray in accordance with Federal Test Method Standard No. 141, Method 6061, except that the panels shall be inclined at an angle of approximately 6 degrees from vertical. Class A and C panels shall be exposed for 5000 to 5500 hours. Class E panels shall be exposed for 2000 to 2500 hours. Panel evaluation after exposure shall include adhesion in accordance with 4.7.8.
- 4.7.12 **Heat Resistance**
- 4.7.12.1 **Long Term Exposure** - One set of each class of panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be placed in a mechanical convection oven and maintained at $300\text{ F} \pm 5$ ($149\text{ C} \pm 3$) for a minimum of 3500 hours. The panels shall be removed, cooled to standard conditions, and bent rapidly over a 4 inch diameter mandrel which has been conditioned at standard temperature. Panel evaluation after bending shall include adhesion in accordance with 4.7.8.

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4.7.12.2 Short Term Exposure - One set of each class of panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing the panels shall be placed in a mechanical convection oven and maintained at $350\text{ F} \pm 5$ ($177\text{ C} \pm 3$) for 70 hours ± 1 . The panels shall be removed, cooled to standard conditions, and bent rapidly over a 4 inch diameter mandrel which has been conditioned at standard temperature. Panel evaluation after bending shall include adhesion in accordance with 4.7.8.

4.7.13 Fluid Resistance

4.7.13.1 Room Temperature - Class A, C, and E panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be tested in accordance with Federal Test Method Standard No. 141, Method 6011, using the fluids and exposure times specified in 3.4.4.1. Panel evaluation after exposure shall include adhesion in accordance with 4.7.8.

4.7.13.2 Elevated Temperature - Class A, C, and E panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be tested in accordance with Federal Test Method Standard No. 141, Method 6011, using the fluids and exposure times specified in 3.4.4.2. Panel evaluation after exposure shall include adhesion in accordance with 4.7.8.

4.7.14 Low Temperature Resistance - Class D panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be aged at room temperature for 16 hours ± 2 after which they shall be placed in a cold box and maintained at $-70\text{ F} \pm 5$ ($-57\text{ C} \pm 3$) for 5 hours. Immediately after removal from the cold box, the panels shall be rapidly bent over a 2 inch diameter mandrel which has been conditioned at the same temperature. Panel evaluation after bending shall include adhesion in accordance with 4.7.8 and examination for checking or cracking or both visible with 10X magnification.

4.7.15 Repairability - Two sets of Class A and C panels shall be prepared and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be immersed for 168 hours ± 2 in the following fluids.

- a. Distilled water at $75\text{ F} \pm 5$ ($23\text{ C} \pm 3$)
- b. Distilled water at $140\text{ F} \pm 2$ ($60\text{ C} \pm 1$)
- c. TT-S-735, Type III fluid at $75\text{ F} \pm 2$ ($23\text{ C} \pm 1$)
- d. TT-S-735, Type III fluid at $140\text{ F} \pm 2$ ($60\text{ C} \pm 1$)

After exposure, remove, wipe dry with clean dry cheesecloth, then dry 4 hours in an air-circulating oven at $140\text{ F} \pm 2$ ($60\text{ C} \pm 1$). Abrade a small area with 400 grit abrasive sheet to expose the substrate. Clean the panels with TT-T-266 lacquer thinner and brush apply colored chromate chemical treatment (MIL-C-5541) to the exposed substrate. Apply a coat of NAI-1269 epoxy primer to the test area of one set of panels in accordance with Process Specification FP-80. Apply a coat of MIL-P-23377 epoxy primer to the test area of the other set of panels in accordance with MIL-C-22751. Cure repaired panels at room temperature for a minimum of 168 hours and then replace in the same test environment for 168 hours ± 2 . Remove panels and test the repaired area in accordance with 4.7.8.

4.7.16 Compatibility With Sealants

4.7.16.1 Class C panels shall be primed and cured in accordance with 4.6.2 and 4.6.3 and sealant applied in accordance with 4.6.4. After curing, two panels with MIL-S-8802 sealant shall be completely immersed for 7 days at $120\text{ F} \pm 2$ ($49\text{ C} \pm 1$) in TT-S-735, Type III test fluid. The remaining panels shall be completely immersed in a 3 percent aqueous sodium chloride solution for 7 days at $120\text{ F} \pm 2$ ($49\text{ C} \pm 1$).

4.7.16.2 Immersion shall be in screw cap jars and the jars shall be sealed by placing a layer of aluminum foil inside the lids.

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- 4.7.16.3 After completion of the immersion period, the jars shall be removed from the heat source and allowed to return to standard temperature prior to removal of specimens. The test shall be completed within 20 hours after removal of the jars from the heat source, and within one hour after removal of the panels from the test fluids.
- 4.7.16.4 After immersion, 2 one inch wide strips shall be prepared through the cloth and sealant to the metal substrate lengthwise along the panel and continuing completely along the unimpregnated cloth. The loose end of each one inch wide strip in turn shall be clamped in one jaw of a suitable recording tensile testing machine and the adjacent end of the panel shall be fastened in the other jaw as shown in Figure 2.
- Cut through the sealant under the cloth, so that an initial separation of the sealant from the panel is promoted.
 - The pull of the cloth shall be at an angle of 180 degrees and a jaw separation rate of 2 inches per minute.

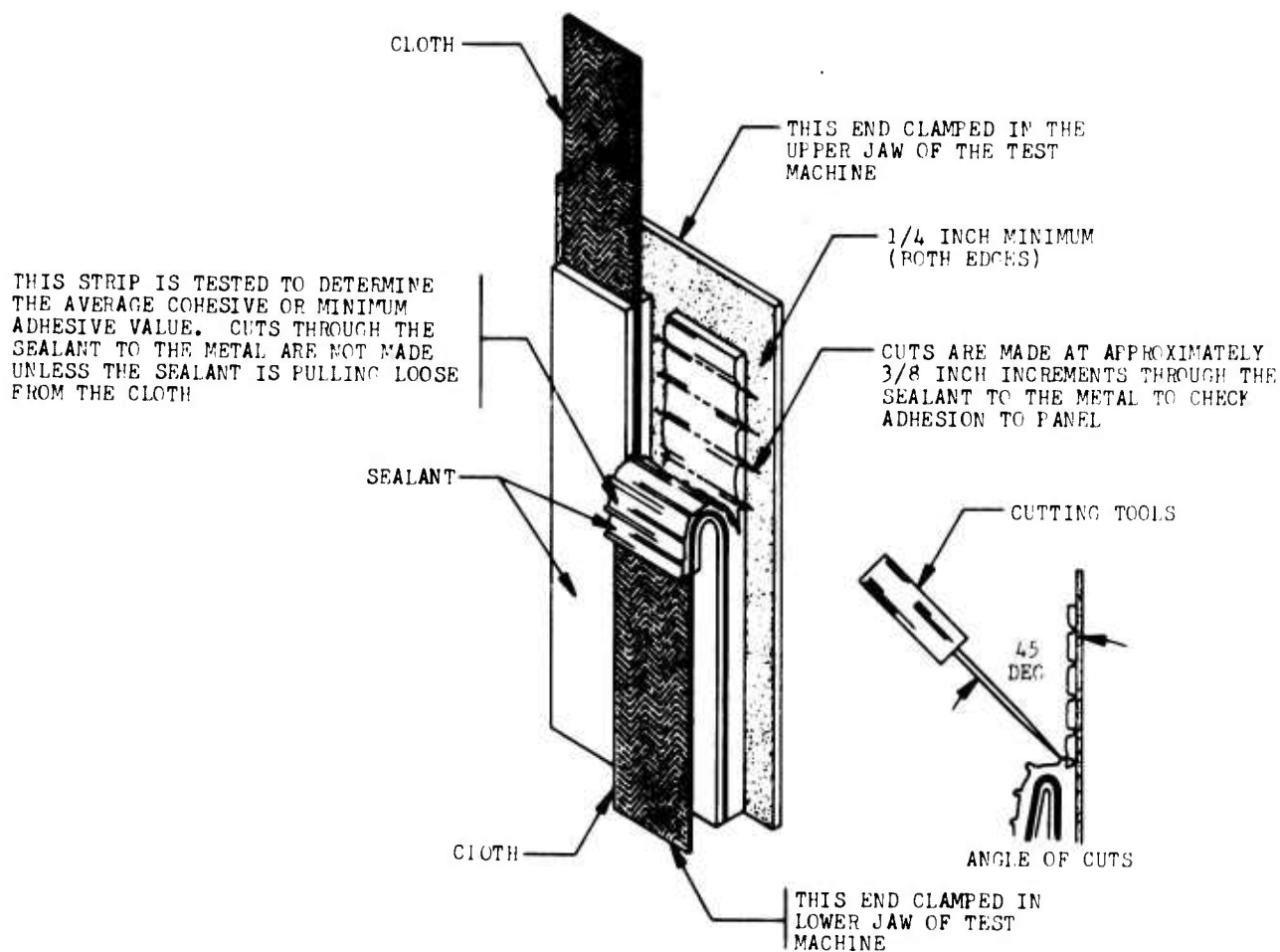


FIGURE 2. COMPATABILITY WITH SEALANT TEST

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- 4.7.16.5 On one of the strips on the panel, cuts in the sealant to the metal panel at the junction of separation shall be made at an angle of 45 degrees towards the direction of separation at approximately 0.375 inch increments (approximately every 24 seconds) as shown in the diagram. (No cuts are required for 100 percent adhesive failure.) On the other strip, except for the initial cut to promote separation, cuts shall be made only as necessary to prevent the cloth from peeling from the sealant. The percent cohesive failure calculated from the ratio of area of cohesive separation to total area of cohesive and adhesive separation from the metal on both sides of the panel shall be determined. The cohesive strength shall be determined during cohesive tear failure.
- 4.7.16.6 The average cohesive strength shall be determined. (Values recorded during cutting shall not be included.)
- 4.7.17 Finish System Compatibility - When tested as specified below, the system shall be considered compatible for use with any of the following: epoxy primers NAI-1269 and MIL-P-23377, topcoated with polyurethane NAI-1278 or 1290 or any government specification topcoat, TT-L-32, MIL-C-22750, in any of the approved colors.
- 4.7.17.1 Class A, C and E panels shall be primed and cured in accordance with 4.6.2 and 4.6.3. After curing, the panels shall be aged at room temperature for 16 hours + 2 after which they shall be cleaned by thoroughly scrubbing with cheesecloth wet with MEK (TT-M-261) or naphtha (TT-N-95). Cleaned panels shall be topcoated within 8 hours after cleaning.
- 4.7.17.2 Apply a single coat of lacquer TT-L-32 to each class of panel at a dry film thickness of 0.4 to 0.5 mil.
- 4.7.17.3 Apply a tack coat of gloss epoxy MIL-C-22750 to each class of panel. Allow to air dry approximately 30 minutes and overcoat with a single wet coat to a dry film thickness of 0.8 to 1.2 mils.
- 4.7.17.4 Apply a single wet coat of polyurethane NAI-1278 topcoat to each class of panel at a dry film thickness of 1.0 to 1.5 mils.
- 4.7.17.5 Allow topcoats to cure 7 days at room temperature before testing the interface adhesion of the topcoat to the primer in accordance with 4.7.8b, c, and d.
- 4.7.17.6 There shall be no removal of the topcoat from the primer.
- 4.7.18 Chemical Properties - Chemical properties shall be determined by spectrophotometer during qualification and shall be controlled on a batch-to-batch basis in accordance with qualification results. The following apparatus and procedures shall be used.
- 4.7.18.1 Apparatus
- a. Infrared spectrophotometer
 - b. KBr, crystal
 - c. Crystal holder
 - d. Circulating air oven
 - e. Aluminum weighing dish
 - f. Glass stirring rod
 - g. Acetone
- 4.7.18.2 Procedure
- a. Mix equal parts primer base with acetone. Stir until homogeneous.
 - b. Transfer homogeneous mixture to KBr crystal with glass stirring rod.
 - c. Place KBr crystal in circulating air oven for 10 minutes at 207 F (97 C).
 - d. Remove KBr crystal from oven, allow to cool to ambient temperature.
 - e. Spread sample to a uniform thickness on the KBr crystal.
 - f. Mount KBr crystal in holder, place holder in IR spectrophotometer, scan with normal slit at fast scan speed.

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4.7.19 Reinspection - All materials shall be reinspected in accordance with 4.4 after one year from date of manufacture, unless all of the batch has been used. If the material passes reinspection test, the usable shelf life shall be extended 6 months. Any materials failing reinspection tests or exceeding the extended shelf life shall be discarded.

4.8 Rejection

- 4.8.1 Any material not conforming to the requirements of this specification shall be rejected.
- 4.8.2 Material rejected in accordance with 4.8.1 may be retested once. For retest, the number of specimens shall be twice the number required by the applicable test method in 4.7. If any retest specimen fails to meet the requirement, the entire batch shall be rejected.
- 4.8.3 No material shall be accepted for delivery that is older than 5 months from date of manufacture.
- 4.9 Reports - Unless otherwise specified, the manufacturer shall furnish with each shipment 3 copies of a report of the results of tests for compliance to the requirements of 4.4 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Primer - Each container shall be durably, conspicuously, and legibly marked with the following:

- a. NAI-1330 primer

NOTE: When the manufacturer's label lists other specifications in addition to NAI Specifications, the NAI designation shall be of the same size and type used in listing the other specifications.

- b. Manufacturer's name and formula number
- c. Batch number
- d. Date of manufacture
- e. Purchase order number
- f. Quantity
- g. Caution note as follows:

CAUTION: This primer must be mixed in accordance with the applicable Northrop Corporation, Aircraft Division Specification prior to use.

5.2 Packaging

- 5.2.1 Packaging shall be such as to assure safe delivery. Each container shall be durably and legibly marked with the following information:
- a. NAI-1330, Revision A Primer
- b. Date of manufacture
- c. Manufacturer's name
- d. Batch number
- e. Purchase order number
- f. Quantity
- g. The unit size as specified on the purchase order

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5.2.2 This material shall normally be available in 55 gallon drums. Larger sizes may be specified on the purchase order.

6. NOTES

- 6.1 Information pertaining to this specification may be obtained from Materials Engineering (3495/31), Aircraft Division.
- 6.2 Suppliers may obtain information pertaining to, or additional copies of, this specification from Northrop Corporation, Aircraft Division, Materiel and Procurement Department (6000/71), 2031 E. Mariposa Ave., El Segundo, California 90245.

RESPONSIBLE ENGINEER <i>T. J. Kissling</i>	NORTHROP Northrop Corporation Aircraft Division QUALIFIED PRODUCTS LIST	NOR-QPL-1330 DATE <u>17 October 1975</u> RELEASE EO <u>E08722</u> CODE IDENT NO 76823
AIRCRAFT DIVISION <i>E. C. Stull</i>		
SPECIFICATION CONTROL <i>B. K. Scher</i>		
PROJECT OFFICE <i>E. J. Anderson</i>		

TITLE:

 ELECTRODEPOSITED CORROSION INHIBITING,
 FLUID RESISTANT PRIMER

PRODUCTS QUALIFIED UNDER SPECIFICATION NAI-1330

Manufacturer's Designation	Manufacturer's Name and Address	Qualification	
		Code Number	Date
Powerclad Green Primer H74GC5 (Initial Change Material)	The Sherwin-Williams Co., Chicago, Illinois	3	10-10-75
	Weight Per Gallon: 9.5 to 10.5 lbs Chemical Properties: See Figure 1 for spectrophotometer curve		
Powerclad Green Primer H74GC6 (Feed Stock)	The Sherwin-Williams Co., Chicago, Illinois	3	10-10-75
	Weight Per Gallon: 10 to 11 lbs Chemical Properties: See Figure 2 for spectrophotometer curve		

(continued on pages 2 and 3)

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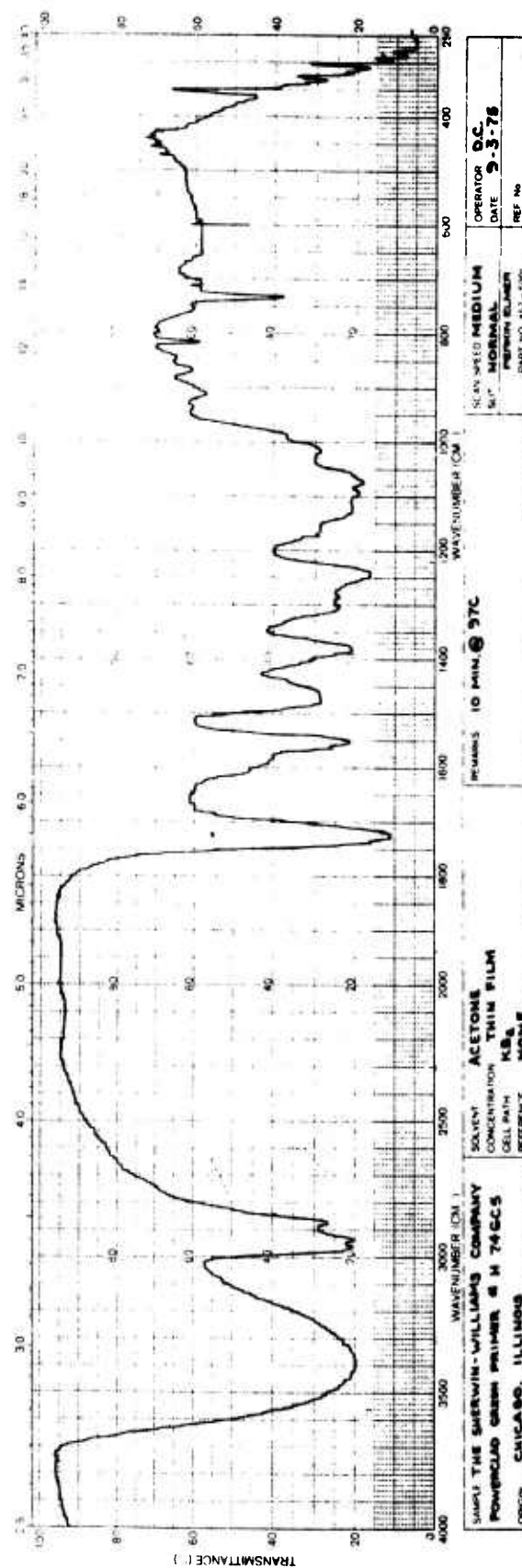


FIGURE 1. CHEMICAL PROPERTIES FOR H746C5

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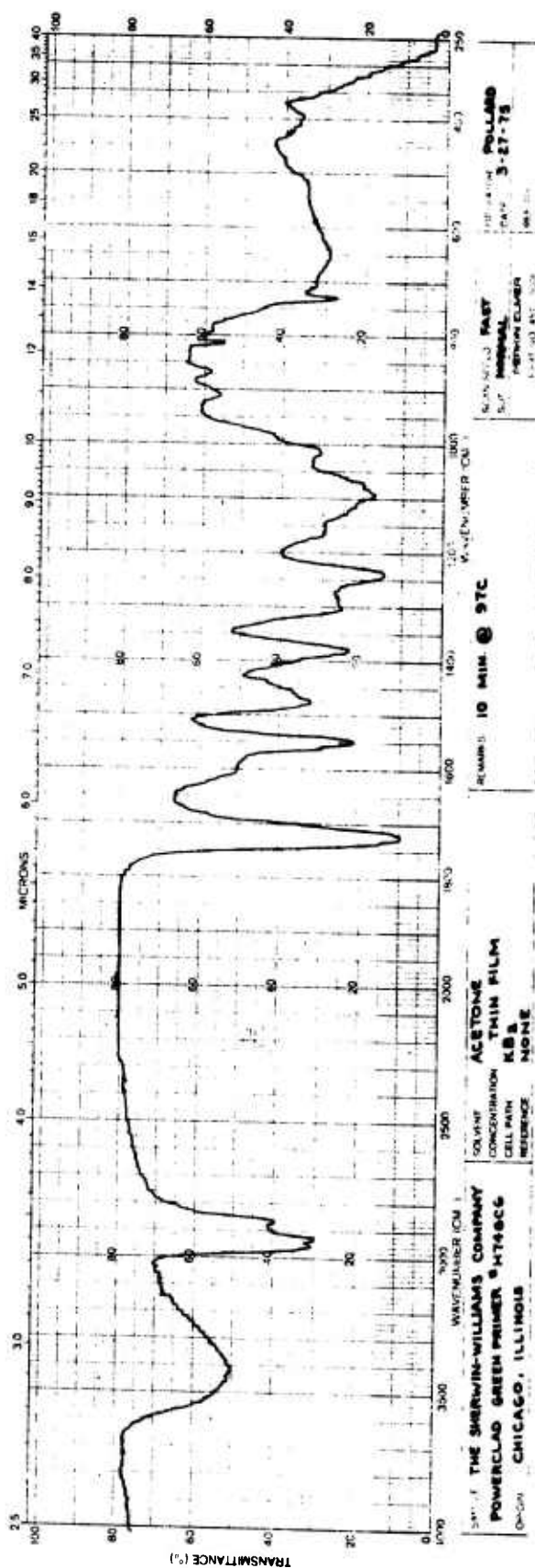


FIGURE 2. CHEMICAL PROPERTIES FOR H74GC6

ADHESION (WET) TAPE TEST

1. SCOPE

1.1 This method covers a procedure suitable for establishing acceptability of intercoat and surface adhesion of an organic coating system immersed in water. This procedure may be utilized as a production test.

2. APPARATUS

2.1 Masking tape. The masking tape used shall be 1-inch wide strip that when tested by method 10, Fed. Test Method Std. No. 147, shall require 45 ± 3 lb. to remove (see 5.1).

2.2 Roller. A 4½-pound rubber covered roller, having a surface Durometer hardness value within the range of 70 to 80, shall be used. The roller shall be approximately 3½ inches in diameter and 1¾ inches in width.

3. REAGENTS

3.1 Distilled water.

4. PROCEDURE

4.1 Apply the coating system to the sub-

strate and allow to dry as specified in the product specification. Immerse the test specimen in distilled water for 24 hours. Remove the test specimen from the water and wipe dry with a soft cloth. Within 1 minute after removal from the water make two parallel scratches, one inch apart, through the coating to the metal with a stylus. Immediately apply a 1-inch wide strip of the masking tape with the adhesive side down across the scratches. Press the tape tightly against the surface of the coating by passing the roller across the tape eight times. Remove the tape with one quick motion and examine for damage to the intercoat or surface adhesion.

5. NOTES

5.1 Minnesota Mining Co. Code No. 250 meets this requirement.

6. PRECISION

6.1 No data.

FLEXIBILITY (PERCENT ELONGATION)

1. SCOPE

1.1 This method covers two procedures for determining the percentage of elongation of single films of organic coating materials when attached to flat steel sheet of uniform surface texture.

2. APPARATUS

2.1 Conical mandrel. The conical mandrel test assembly illustrated in figure 1.

2.2 Cylindrical mandrels. A series of straight, smooth, cold rolled steel rods having diameters of $\frac{1}{8}$, $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$, and 1 inch and held firmly by suitable supports.

2.3 Metal panels.

2.3.1 The panels shall be cut from S.A.E. No. 1010, finish A strip or sheet steel having a Rockwell "B" hardness not exceeding 50. The dimensions of the individual panel shall not exceed $4\frac{1}{2}$ inches in width, $7\frac{1}{2}$ inches in length and $\frac{1}{32}$ -inch in thickness. The panels shall be thoroughly cleaned just before use by whichever of the procedures described in 4.1 of 2011.1 that is applicable. The edges of the panels shall be slightly rounded to remove all burrs.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of test specimens.

4.1.1 Application. Prepare at least three test specimens by applying the coating material at the thickness and in the manner specified in the product specification to one of the metal panels. To a second panel apply the coating having a thickness of not more than 0.5 mil less than that specified and to a third panel apply a coating having a thickness of not more than 0.5 mil greater than that specified.

4.1.2 Drying. Unless otherwise specified, dry the test films by whichever of the following procedures that is applicable.

4.1.2.1 Air drying materials. Air dry at 21° to 32° C. (70° to 90° F.) for 72 hours and then bake for 4 hours at $121^{\circ}\pm 2^{\circ}$ C. ($250^{\circ}\pm 4^{\circ}$ F.).

4.1.2.2 Baking materials. After reasonable period at room temperature for solvent release,

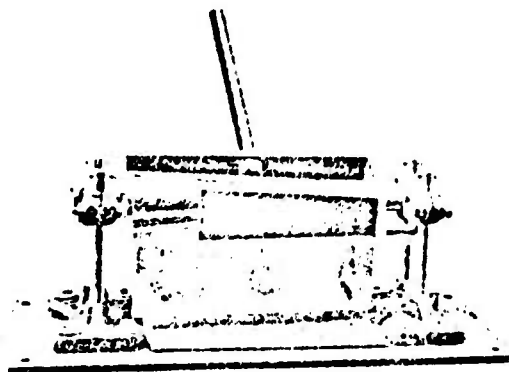


FIGURE 1. Conical mandrel test apparatus.

bake for 1 hour at $121^{\circ}\pm 2^{\circ}$ C. ($250^{\circ}\pm 4^{\circ}$ F.), air dry for 24 hours at 21° to 32° C. (70° to 90° F.) and then bake for 4 hours at $121^{\circ}\pm 2^{\circ}$ C. ($250^{\circ}\pm 4^{\circ}$ F.).

4.2 Performance of test. Condition the test specimens at $23^{\circ}\pm 1.1^{\circ}$ C. ($73.4^{\circ}\pm 2^{\circ}$ F.), and 50 ± 4 percent relative humidity for 24 hours, determine the thickness of each test film (method 6181 or 6183), and then bend the panels and calculate the percentage of elongation of the coating by one of the following procedures.

4.2.1 Conical mandrel. With the operating lever of the apparatus in a horizontal position, slip the test specimen between the mandrel and the drawbar with the test film towards the drawbar. Place two sheets of No. 1 Kraft wrapping paper, substance 30, thoroughly lubricated on each side with talc, between the test film and the drawbar. Rigidly clamp the test panel in a vertical position adjacent to the mandrel by placing the long edge behind the clamping bar in such a manner that the panel will be set up to the narrow end of the mandrel. Now move the lever at a uniform rate so as to bend the test specimen approximately 180° in about 15 seconds. Care must be taken at the end of the stroke so as not to unnecessarily deform the specimen. Immediately examine the bent area for cracking of the test film using 7-power mag-

FED. TEST METHOD STD. NO. 441a

nification under the conditions of illumination prescribed as follows: Examine the film at the bend under a magnification of 7 diameters, using diffused light having a color temperature of 6,500° Kelvin and an intensity of about 165 foot-candles. (For those not equipped with a standard illuminant, viewing the test coating at a north window illuminated by a fairly light overcast sky will approximate these conditions.) Mark and measure the extent of the first continuous crack, starting from the small end of the mandrel, which shall be considered the end point. Return the drawbar to the starting position and remove the test specimen. Using the length of the crack, determine the percent elongation of each test film from the curve shown in figure 2, making the necessary correction for the thickness of each test film as determined from the curve shown in figure 3 according to the following example.

4.2.1.1 Example. Suppose that examination of the test film on the bent cold rolled steel specimen $\frac{1}{32}$ inch (see 5.1) in thickness shows that the end of the first crack in the test film is at a distance of 5 inches from the base of the cone. It is merely necessary to refer to figure 2 and to read directly the percentage of elongation of the test film at the designated distance from the base of the cone. A correction for thickness of test film shall be added to the elongation as determined from figure 3. If the test film thickness were 0.002 inch, from figure 2 the elongation at 5 inches from the base of the cone is 5.2

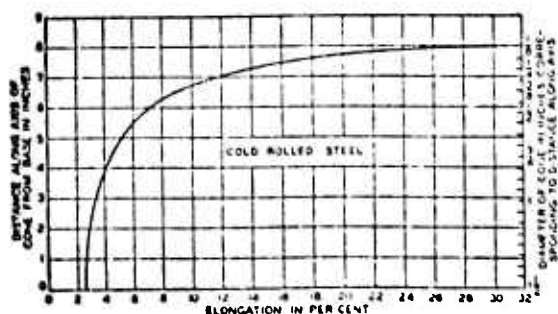


FIGURE 2. Distance along cone and corresponding mandrel size versus percentage elongation for specimens on cold-rolled steel $\frac{1}{32}$ inch in thickness.

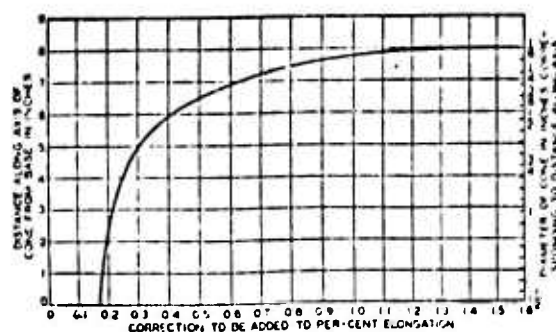


FIGURE 3. Correction for thickness of film.

percent. From figure 3 the correction to be added per 0.001 inch of test film is 0.305 percent at 5 inches from the base of the cone. Thus when the total test film thickness is 0.002 inch the actual percentage elongation at 5 inches is $5.2 \pm 2 (0.305)$ or 5.8 percent.

4.2.2 Cylindrical mandrels. Place the test specimens, with the coated side uppermost, on a mandrel of the specified diameter, at a point equally distant from the top and bottom edges of the panel, and bend the panel double in approximately 1 second. Examine the film at the bend under a magnification of 7 diameters, using diffused light having a color temperature of 6,500° K. and an intensity of about 165 foot-candles. (For those not equipped with a standard illuminant, viewing the test coating at a north window illuminated by a fairly light overcast sky will approximate these conditions.) Use different diameter mandrels until the test film of each indicated thickness (see 4.1.1) shows cracking. Record the diameter of the mandrel where no cracking occurs in each case.

4.2.2.1 Calculations. Calculate the percent elongation of each test film by the following formula:

$$\text{Percent elongation} = 100 \times \frac{t}{d+t}$$

where: t = Thickness of panel.
 d = Diameter of mandrel.

4.2.2.1.1 A correction must be added to the calculated value to allow for the thickness of the test film in each case. The correction table for use on cold-rolled steel is as follows:

TABLE I. Diameter of mandrel (d)

1 inch	¾ inch	½ inch	¼ inch	⅛ inch	⅙ inch
0. 21	0. 26	0. 38	0. 50	0. 73	1. 37

422.12 This correction is per mil of test film. If the test film is more or less than one mil in thickness, the correction added is greater or less according to the expression :

$$\text{Correction} = fc$$

where: *f*=Film thickness of coating in mils.

c=Chart correction for 1 mil.

4.3 Reporting of results.

4.3.1 Report the percentage elongation of

the material at the specified film thickness, as interpolated from the percentages of elongation calculated for the three different test film thicknesses.

5. NOTES

5.1 Other suitable base materials for the test specimen may be employed. The method for calculating the elongation of organic coatings applied to base materials other than the steel specified herein is given in the Appendix to ASTM D522-41, Standard Method of Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus.

6. PRECISION

6.1 No data.

SALT SPRAY (FOG) TEST

1. SCOPE

1.1 This method sets forth the conditions required in salt spray (fog) testing for specification purposes. The test specimens may be exposed to either 5 or 20 percent salt (NaCl) spray as specified in the product specification. When the salt concentration is not stated in the specification, the 20 percent solution shall be used.

2. REAGENTS

2.1 Salt solution. Either 5 or 20 percent sodium chloride solution, as stipulated (see 1.1) meeting the following requirements:

2.1.1 *Ingredients.*

2.1.1.1 *Sodium chloride.* The sodium chloride shall be substantially free of nickel and copper and contain, on a dry basis, not more than 0.1 percent of sodium iodide and not more than 0.3 percent of total impurities.

2.1.1.2 *Water.* Distilled water or water containing not more than 200 parts per million of total solids.

2.1.2 *Concentration.*

2.1.2.1 *Five-percent solution.* Dissolve 5 ± 0.5 parts of the sodium chloride in 95 ± 0.5 parts by weight of the water.

2.1.2.2 *Twenty-percent solution.* Dissolve 20 ± 2 parts of the sodium chloride in 80 ± 2 parts by weight of the water.

2.1.3 *Properties.*

2.1.3.1 *Specific gravity.* The specific gravity of the 5-percent solution shall be between 1.0268 and 1.0413 and that of the 20-percent solution between 1.126 and 1.157, when measured between 92° and 97° F.

2.1.3.2 *pH value.* The pH value of each solution shall be such that when atomized at 95° F., the collected solution will be in the pH range of 6.5 to 7.2 (see 5.1). The pH measurement shall be made electrometrically using a glass electrode with a saturated potassium chloride bridge or colorimetrically using bromthymol blue as the indicator, provided the results obtained by the colorimetric method are equivalent to those obtained in the electrometric

method. Only dilute solutions of A.C.S. reagent grade hydrochloric acid or sodium hydroxide in distilled water shall be used in adjusting the salt solution to the specified pH range.

2.1.4 *Removal of suspended solids.* All solids shall be removed from the salt solution either by filtration before the solution is placed in the reservoir or by placing a double thickness of cheesecloth over the end of the tube leading from the reservoir to the atomizer.

3. APPARATUS

The apparatus used in the salt spray test shall include the following:

3.1 *Exposure chamber.* The exposure chamber shall be of suitable size, construction and design to satisfy the conditions of the test herein described. It shall be equipped with racks or other means for properly supporting the test specimens. The chamber shall be made of material which will not affect the corrosiveness of the fog, such as glass, rubber- or plastic-lined steel, stone, or suitably coated wood. It shall be properly vented and so designed that the drops of solution, which accumulate on its ceiling or cover will not fall on the test specimens, and no liquid that has come in contact with the test specimens will return to the salt solution reservoir.

3.2 *Salt solution reservoir.* A container suitable for storing the salt solution with a pH below 6.5 made of rubber- or plastic-lined steel, stone or glass.

3.3 *Atomizing equipment.* The atomizing equipment shall be of such construction and design that the conditions of atomization and quantity of fog prescribed in 4.2.2 will be maintained throughout the test.

3.3.1 *Atomizing nozzles.* The nozzles shall be made of glass, monel metal, hard rubber or plastic. The orifices of the nozzles shall be kept smooth at all times to insure delivery of a finely divided, wet, dense fog.

3.3.2 *Air supply.* The compressed air supply to the nozzles for atomizing the salt solution shall be free of oil and dirt (see 5.2)

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TABLE I

Air pressure, p.s.i.	12	14	16	18
Temperature, °F.	114	117	119	121

and maintained between 10 and 25 p.s.i. (see 5.3). To avoid any substantial change in the moisture content of the salt fog particles, the air must have a humidity between 95 and 98 percent after release inside the cabinet. Since the relative humidity of compressed moist air drops sharply when it expands without appreciable drop in temperature, such as on release from the nozzles, it is necessary to saturate the air at a temperature higher than that maintained within the chamber. This is accomplished by passing the cleaned compressed air in finely divided bubbles through a saturator of corrosion resistant material (see 5.4) containing heated water of a controlled temperature. Required temperatures for varying values of air pressure are given in table I.

3.4 Preparation of test specimens.

3.4.1 Test panels. Unless otherwise specified, 3- by 6-inch steel panels as prescribed in 1 of method 2011 shall be used. The panels shall be cleaned and prepared by whichever procedure in section 3 of method 2011 is specified in the product specification. The backs and edges of the panels shall be coated with a suitable protective material.

3.4.2 Test film. The manner of application of the material under test, the thickness of the film and the conditions for drying it shall be as specified in the product specification. Unless otherwise specified, the test film shall be scored through to the metal with a sharp instrument in a line starting about one-quarter inch from the top edge, running parallel to and about 1 inch from the long edge, and ending about one-quarter inch from the bottom edge of the panel.

4. PROCEDURE

4.1 Position of test specimens. Unless otherwise specified, arrange the flat specimens, and where practicable other specimens, in such a manner that their dominant test surfaces will be supported or suspended at 15° to 30° from

the vertical and parallel to the principal horizontal flow of the fog through the chamber. Support the test specimen, when possible, from either the bottom or the side. When suspension is necessary, use glass hooks or waxed string (see 5.5) and secondary supports at the bottom of the specimens, if necessary, to hold them in the above specified position. Arrange the test specimens so that they do not contact each other or any material capable of acting as a wick; so as to permit free settling of the fog on all specimens; and so that corrosion products and condensate will not fall from one specimen upon another.

4.2 Conditions in the salt spray chamber.

4.2.1 Temperature. Maintain a temperature of 95° plus 2° or minus 3° F. within the exposure zone of the salt spray chamber, during the entire test. Temperature control may be accomplished by housing the apparatus in a constant temperature room, by thoroughly insulating the apparatus and preheating the air to the proper temperature prior to atomization, or by surrounding the chamber with a jacket containing water of a controlled temperature. Immersion heaters must not be used to maintain the temperature within the exposure chamber. Record the temperature of the exposure zone twice a day at approximately a 7-hour interval (except weekends and holidays). The temperature may be obtained with a continuous recording device or by a thermometer which can be read from outside the closed chamber. The recorded temperature must be obtained with the salt spray chamber closed to avoid a false low reading because of wet bulb effect when the chamber is open.

4.2.2 Atomization and quantity of fog. Arrange or baffle the spray nozzles so that none of the spray will impinge directly on the test specimens. Place two clean fog collectors (see 5.6) within the exposure zone in the proximity of the test specimens but not so near that condensate will drop from the test specimens into them. Where possible one collector should be placed near the nozzle and the other as far away as practicable. Maintain the rate of atomization (see 5.7) and quantity of fog within the

chamber so that for each 80 square centimeters of collecting area from 0.5 to 3.0 milliliters of solution will be collected per hour, based on an average run of at least 16 hours. Remove and determine whether the collected solutions are within the specific gravity and pH ranges prescribed in 2.1.3.1 and 2.1.3.2 for the concentration of the salt solution being used. If such is not the case, make the necessary adjustments before proceeding with the test. Make sufficient periodic collections during the exposure to insure proper atomization. These conditions may have to be modified when chambers having volumes of more than 12 cubic feet are used, in order to meet the requirements for operating conditions.

4.3 Continuity and duration of exposure. Keep the chamber closed and the spray operating continuously, except for the short daily interruptions necessary to inspect the test specimens, and check and replenish the salt solution, for the specified period of exposure or until definite signs of failure are observed. The length of the exposure shall be as specified in the product specification.

4.4 Examination of specimens. At the end of the specified exposure period, examine the test specimens immediately for conformance to the requirements stipulated in the product specification. To facilitate the inspection of the test specimens, gently wash or dip them in clean running water, not warmer than 100° F., to remove any salt deposit, and dry immediately by sponging with a soft cloth or blotting paper. When required, remove the corrosion products by light brushing and observe to what extent the substrate has been affected.

5. NOTES

5.1 Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature. If the water is heated to 95° F. or above to expel the

carbon dioxide immediately before preparing the salt solution and the solution then adjusted to a pH range of 6.5 to 7.2, its pH will not materially change when atomized at 95° F.

5.2 The air supply may be freed of oil and dirt by passing it through a water scrubber or at least 2 feet of suitable cleaning material, such as asbestos, sheep's wool, excelsior, slag wool, or activated alumina.

5.3 Atomizing nozzles may have a "critical pressure" at which an abnormal increase in corrosiveness of the salt fog occurs. If the "critical pressure" of a nozzle has been established, control of fluctuation in air pressure within ± 0.1 p.s.i. by means of a suitable pressure regulator valve, minimizes the possibility that the nozzle will be operated at its "critical pressure".

5.4 The water in the saturator should be changed once a week to permit the removal of impurities of the air.

5.5 All parts that come in contact with the test specimens shall be of materials that will not cause electrolytic corrosion.

5.6 Crystallizing dishes or glass funnels with stems inserted through stoppers into graduated cylinders make suitable collecting devices. Dishes and funnels with a diameter of 10 centimeters have an area of about 80 square centimeters.

5.7 Suitable atomization has been obtained with noncorrosive nozzles in exposure chambers having volumes less than 12 cubic feet under the following conditions:

- (1) Nozzle pressure of from 12 to 18 pounds per square inch.
- (2) Orifices of from 0.02 to 0.03 inch in diameter.
- (3) Atomization of approximately 3 quarts of the salt solution in 24 hours within a 10 cubic foot chamber.

6. PRECISION

6.1 No data.

APPENDIX C

CANDIDATE ELECTROPRIMER FORMULATIONS

DEFINITION OF CANDIDATE ELECTROPRIMER FORMULATIONS

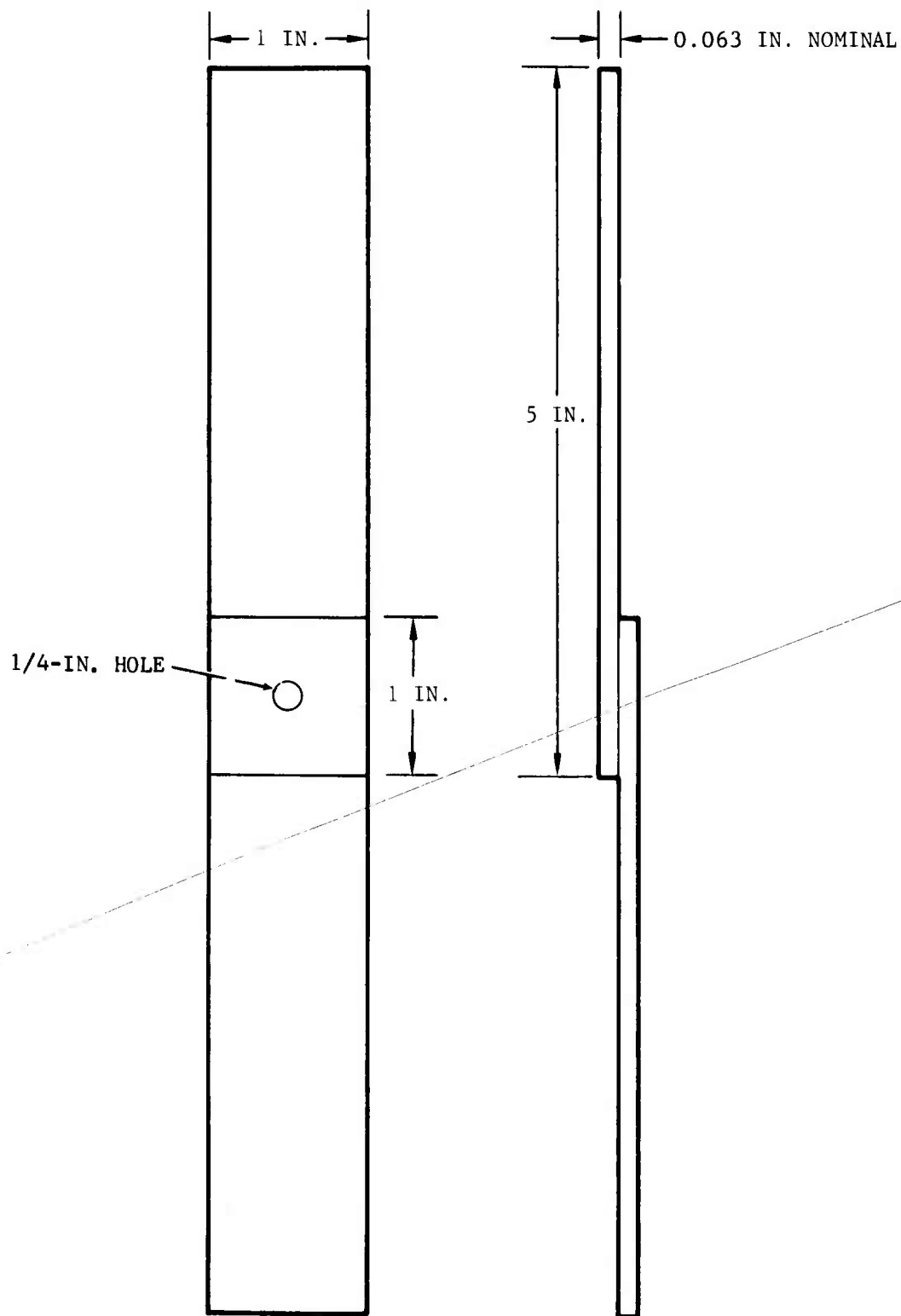
FORMULATION IDENTIFICATION	FORMULATION SEQUENCE	APPLICATION TYPE	APPLICATION REQUIREMENTS (1)		BASE RESIN	CROSS-LINKER (CATALYST)	RESIN SOLUBILIZER	PIGMENT	CORROSION INHIBITOR	COALESCING SOLVENT	NOMINAL PH	NOMINAL SOLIDS (%)	NOMINAL BATH CONDUCTIVITY (OHM-CM)	CURE SCHEDULE	
			VOLTS (DC)	TIME (SEC)										TEMP (°F)	TIME (MIN)
SA 4185	A	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	SrCr ₂ O ₇	(6)	6.8	10	550	275	30
SA 4186	A	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	SrCr ₂ O ₇	(6)	7.3	10	600	275	30
SA 4187	A	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	SrCr ₂ O ₇	(6)	7.5	10	550	275	30
SA 4188	B	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	7.2	10	650	275	30
SA 4190	B	Anodic	30	30	Polyester	(3)	basic (4)	TiO ₂	none	(6)	7.4	10	600	275	30
SA 4491	Control	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	7.1	10	750	275	30
SA 4472	C	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	6.7	10	645	275	30
SA 4473	C	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	7.7	10	615	275	30
SA 4474	C	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	8.2	10	700	275	30
SA 4475	C	Anodic	30	30	Polyester	(2)	basic (4)	TiO ₂	none	(6)	8.5	10	670	275	30
SA 4521	D	Cathodic	50	5	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	6.7	10	950	400	30
SA 4542	E	Cathodic	50	5	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	5.5	10	920	375	30
SA 4708	F	Cathodic	30	15	Acrylic	(3)	acid (4)	TiO ₂	none	(6)	6.7	10	950	325	30
SA 4753	F	Cathodic	30	15	Acrylic	(3)	acid (4)	TiO ₂	SrCr ₂ O ₇	(6)	6.5	10	950	325	30
SA 4777	G	Cathodic	30	15	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	6.5	10	900	375	30
SA 4778	G	Cathodic	30	15	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	6.5	10	900	375	30
SA 4824	H	Cathodic	20	15	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	6.7	16	(7)	350	30
SA 4825	H	Cathodic	20	15	Modified Epoxy	(3)	acid (4)	none	SrCr ₂ O ₇	(6)	6.7	13	(7)	350	30
SA 4826	H	Cathodic	30	15	Modified Epoxy	(3)	acid (4)	TiO ₂	none	(6)	6.7	16	(7)	350	30
SA 4827	H	Cathodic	20	15	Modified Epoxy	(3)	acid (4)	none	SC-1 (5)	(6)	6.7	13	(7)	350	30
SA 4828	H	Cathodic	20	15	Modified Epoxy	(3)	acid (4)	none	SC-1 (5)	(6)	6.5	16	550	345	30
C-5301	I	Cathodic	30	15	Modified Epoxy	(3)	(4)	TiO ₂	none	(6)	6.5	16	550	345	30

NOTES:

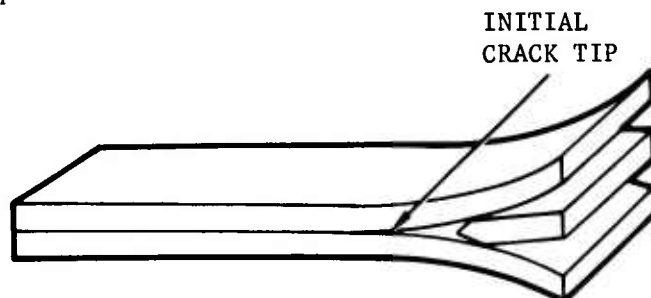
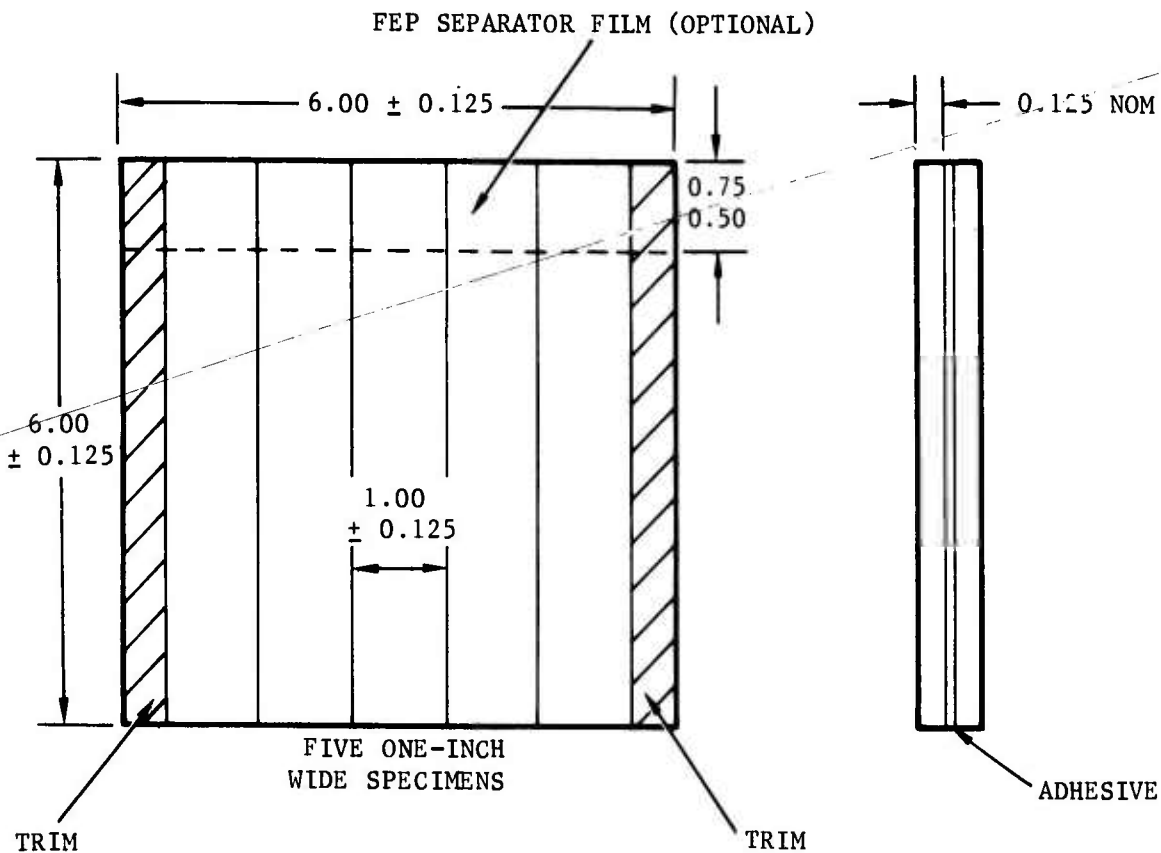
- (1) Approximate time and voltage to produce a 0.2 mil film
- (2) Standard NAI 1330 Polyester catalyst
- (3) Experimental cross-linker
- (4) Sherwin-Williams experimental
- (5) Nalzin SC-1, product of National Lead Company
- (6) Sherwin-Williams experimental
- (7) Not determined

APPENDIX D

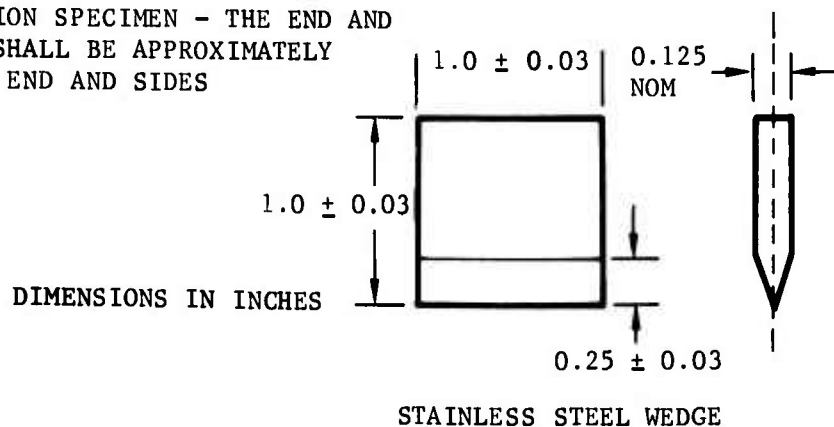
TEST SPECIMEN CONFIGURATIONS AND ADHESIVE
BONDING LAY-UP AND CURE CONTROL



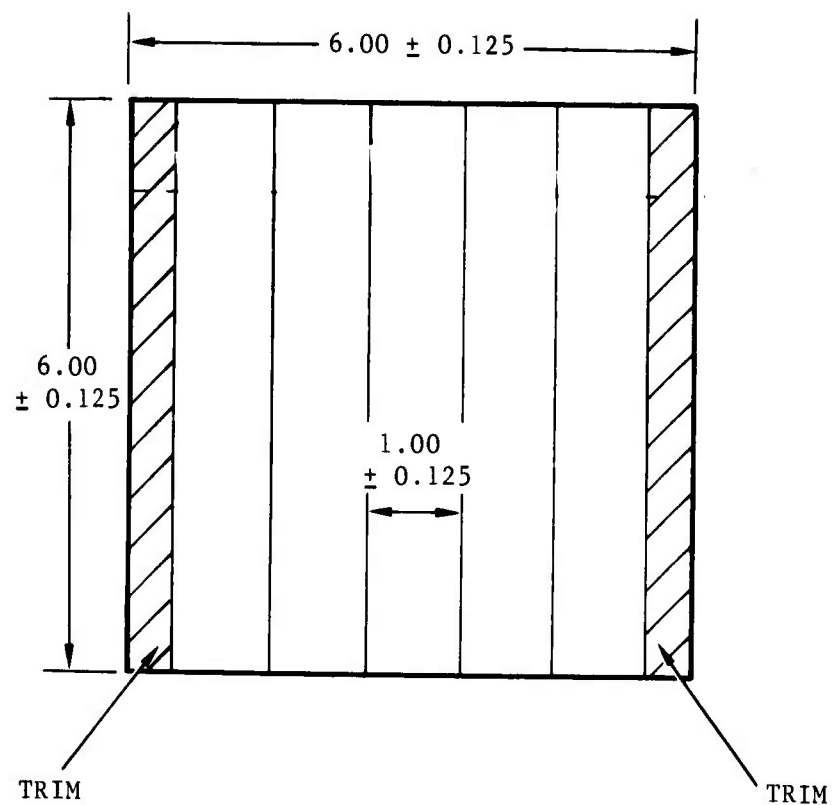
SCREENING TEST STRESS RUPTURE SPECIMEN



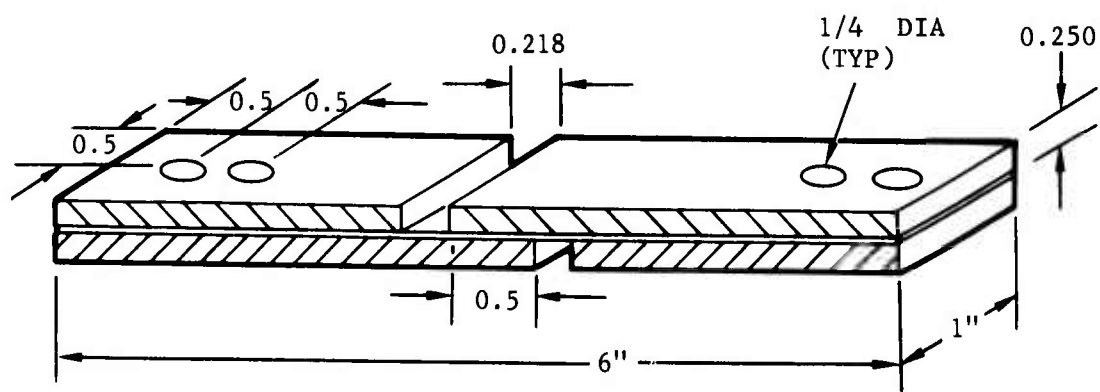
WEDGED CRACK EXTENSION SPECIMEN - THE END AND SIDES OF THE WEDGE SHALL BE APPROXIMATELY FLUSH WITH SPECIMEN END AND SIDES



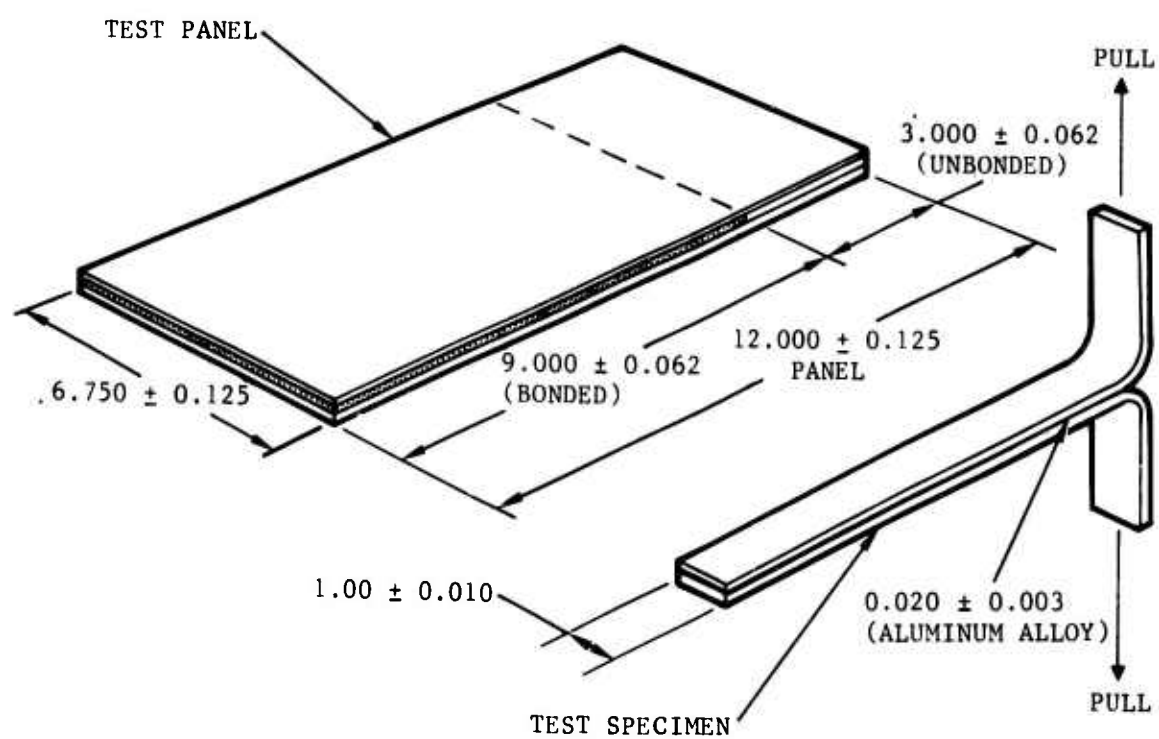
STANDARD WEDGE TEST PANEL AND COUPON



DIMENSIONS IN INCHES



THICK ADHEREND MACHINED TENSILE SHEAR AND STRESS RUPTURE SPECIMEN



DIMENSIONS IN INCHES

T-PEEL TEST SPECIMEN

ADHESIVE BONDING LAY-UP AND CURE CONTROL

SPECIMEN LAY-UP PROCEDURE FOR ADHESIVE BONDING

The primed and cured panels were assembled into pairs using one layer of FM-123-2, 0.085 psf film adhesive applied to one faying surface of one panel for each pair. The faying surfaces of each pair were mated carefully to avoid entrapment of air in the bondline. Care was taken to insure that the paired adherends were matched and square, i.e., the edges were aligned without overlaps. Thermocouples were installed into the bondline and taped to prevent pull out. One layer of nonporous Teflon release film (Armalon) was placed over the assembled panels. Three layers of vacuum transfer cloth were applied in the same manner. The assembled panels were placed into a nylon vacuum bag with a vacuum source and the vacuum bag was then sealed. The vacuum bag was leak checked before autoclave cure.

ADHESIVE CURE CONTROL FOR BONDED SPECIMENS

All adhesive cures of test specimens were accomplished in a 5 foot diameter autoclave equipped with Research Inc. Data Trak programmers. The Data Trak programmers provide automatic temperature and pressure control. A programmed standard heat up rate of 3-5°F/minute was used for all adhesive cures. All adhesive bonds were maintained at the required adhesive cure temperature for 90 minutes with 40 psi augmented pressure, and the vacuum bag vented to atmosphere. The cool down rate to 150°F was 8-10 °F/minute before venting augmented pressure. The programmed Data Trak cure cycle was used on all contract adhesive cures to insure uniformity of the bonding process throughout the work effort.

APPENDIX E
DEFINITION OF TERMS

DEFINITION OF TERMS

ANTIOXIDANT (CORROSION INHIBITOR) — A material added to the electroprimer to retard oxide changes after surface preparation, e.g., lead chromate, zinc chromate and strontium chromate. In retarding surface oxide changes, antioxidants promote adhesive bond durability.

PIGMENT — An inorganic material added to the electroprimer to provide opacity to the deposited film, e.g., titanium dioxide, aluminum oxide, cadmium dioxide.

COLORING AGENT — A material added to the electroprimer specifically to provide color in the deposited resin film, e.g., "iron-yellow".

COALESCENCE (FILM) — The ability of deposited electroprimer resin to flow after deposition to provide a continuous, uniform film.

CROSSLINKER (CATALYST) — The chemical substance in the electroprimer formulation which provides the crosslinking bonds between the basic resin system molecules. The crosslinker becomes an integral part of the thermoset macromolecule.

FEATHERING (OXIDE) — The appearance of the aluminum oxide when the oxide grows in a random loose powdery manner as opposed to the tight dense appearance of a columnar deposited oxide.